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FOLLOWING
NOTE

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APPENDIX NUMBER I HAS BEEN OMITTED FROM THIS FICHE BECAUSE IT IS OF POOR REPRODUCTIVE QUALITY

L'APPENDICE NUMÉRO I A ÉTÉ OMIS DE CETTE FICHE ÉTANT DONNÉ SA PIÈTRE QUALITÉ DE REPRODUCTION

OFFSHORE PRODUCT QUALITY
PHASE II

FINAL PROJECT REPORT

DSS Contract Number: 23440-7-9221/01-SQ

Science Branch Contracting Officers: D. Fraser

CANMET Scientific Authorities: G. Feasby

Kalium Chemicals Project Leader: J. A. Gosselin

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MICROMEDIA

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EXECUTIVE SUMMARY

The purpose of this project is to improve the quality of potash products supplied by Canpotex, the Saskatchewan potash producers marketing agency, to offshore customers. This project is a continuation of the work which was conducted by the Potash Corporation of Saskatchewan in 1987. The majority of the work which has been reported in this study will be continued and/or completed in the third phase of this project. Co-operation between Saskatchewan potash producers has resulted in a review of the quality assurance program for offshore shipments, the continued development and standardization of measures of product quality, the development and evaluation of equipment for monitoring the effect of environmental conditions on potash piles, and a better understanding of how potash materials degrade and/or cake through the use of scanning electron microscopy (SEM).

A review of the quality assurance program for offshore shipments has indicated that significant improvements could be made in all areas. A tour of the Neptune Bulk Terminal in Vancouver revealed that the handling and sampling of product could significantly be improved. Recommendations were made to Canpotex on how to improve the handling and sampling procedures at Neptune. Sampling equipment and procedures for sampling individual railcars was developed by Kalium. Similar sampling equipment was also fabricated for sampling at offshore locations. The sample preparation and analysis procedures used by Canpotex's quality assurance representatives, Pkb Scania were also reviewed. Recommendations on how to improve sample preparations were made. A task force consisting of quality control representatives for each minesite was formed. The task force is presently in the process of comparing the AOAC standard procedures used by Pkb Scania with the various procedures used by each minesite. In order to ensure the development of a proper quality assurance program for offshore shipments it was recommended that Canpotex seriously consider adding a quality assurance expert to their staff.

A dedusting test which was developed by the Potash Corporation of Saskatchewan (PCS) in the first phase of this study was adopted has a standard by Saskatchewan potash producers. A dedusting apparatus was fabricated for each minesite and plans are for round robin testing of the new dedusting test. Other destructive product quality tests were evaluated such as the moisture absorption test developed by International Mineral Corporation of Canada (IMCC). The IMCC moisture absorption test provided preliminary information on the amount and rate of moisture absorption of various potash products exposed to different relative humidities. Initial work was also started in developing methods of non-destructive testing for compacted potash flake. The initial work on impact echo showed that good and poor quality flake had different sound patterns, while the light reflectance work showed that good flake reflected less light than poor flake. These destructive and non-destructive measures of quality could have a significant impact on the potash producers ability to manufacture quality products, especially compacted products.

PCS developed a pilot scale humidity and temperature controlled environment which can be used to store and expose a pile of potash to different atmospheric conditions. The effect these atmospheric changes had on the potash pile were monitored by using a series of humidity and temperature probes which were placed at different depths in the pile. The data being collected from these probes combined with the analyses being performed on the potash samples taken through the experiment, will provide a better understanding of the effect environmental conditions have on a potash pile.

PCS evaluated scanning electron microscopy (SEM) as a method for gaining a better understanding of how product which has been exposed to the PCS degradation test breaks down. Samples which were exposed to the PCS caking test were also evaluated with the SEM to determine the bonding mechanism at the particle to particle contact points. Scanning electron microscopy has shown to be a very useful tool for understanding the micro structure of potash products and may be used in future work.

INTRODUCTION

The purpose of this study was to improve the quality of Saskatchewan potash products delivered to offshore markets.

Over supply and a trend towards coarser grades of potash have pressured potash producers into improving the quality of their products in order to maintain market share. For Saskatchewan potash producers to maintain or increase their competitiveness they must produce products of equal or better quality than offshore competitors (USSR, German Democratic Republic, Israel and Jordan).

Offshore customers require products which meet chemical and particle size distribution specifications. The products must also have good handling (free flowing, dust free) and storage (durable, no caking) characteristics. Canpotex, the producer-owned potash marketing agency, presently markets 18 grades of potash.

There are basically three types of potash material produced by Saskatchewan potash producers. The first is the natural crystal from the ore deposit, the second is a crystalline material produced in a crystallization process and the third is a compacted material which is produced by taking the smaller size fraction of the first two types of potash and compressing the material at high pressures to produce a sheet of potash which is crushed and screened to the desired size. These potash materials are sometimes mixed together to form a final product.

This study is a continuation of the work which was conducted by the lead company the Potash Corporation of Saskatchewan (Offshore Product Quality, DSS Contract Number: 045Q.23440-6-9122). Although the development of new and improved measures of product quality is also an important part of this study, the scope of the study was expanded to include the following:

- a complete review of the quality assurance program for offshore shipments.

- an attempt to develop an understanding of the effects changes in atmospheric conditions have on product competence and storage characteristics.
- the evaluation of scanning electron microscopy as a tool to gain a better understanding of the bonding and degradation mechanisms.

SPPA - OFFSHORE PRODUCT QUALITY: PHASE II

QUALITY ASSURANCE PROGRAM FOR OFFSHORE SHIPMENTS
(SUB-PROJECT 1224.01)

PROGRESS REPORT

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May, 1988

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TASK ONE: EVALUATION OF QUALITY ASSURANCE PROGRAM

INTRODUCTION

The majority of Saskatchewan potash shipped to export markets is warehoused and/or directly loaded into ocean-going vessels at Neptune Bulk Terminal Ltd. (Appendix I) and Vancouver Wharves in Vancouver, B.C. (Appendix II). Since the majority of the Granular (compacted) material was shipped through Neptune Terminal, efforts were concentrated on this facility. The sampling and analysis of all offshore shipments is performed by Pkb Scania, Cargo Surveyors Ltd., (212 Brooksbank Avenue, North Vancouver, B.C.). This company also performs draft surveys and sampling at the destination point of each shipment, if required.

On October 28, 1987 Dave Dusterbeck, P. Eng., Process Engineer and Joe Gosselin, Research Chemist, Kalium Canada Ltd. evaluated the handling, warehousing and sampling performed at Neptune Terminals. The sampling, sample preparation and analyses performed by Pkb Scania were also evaluated.

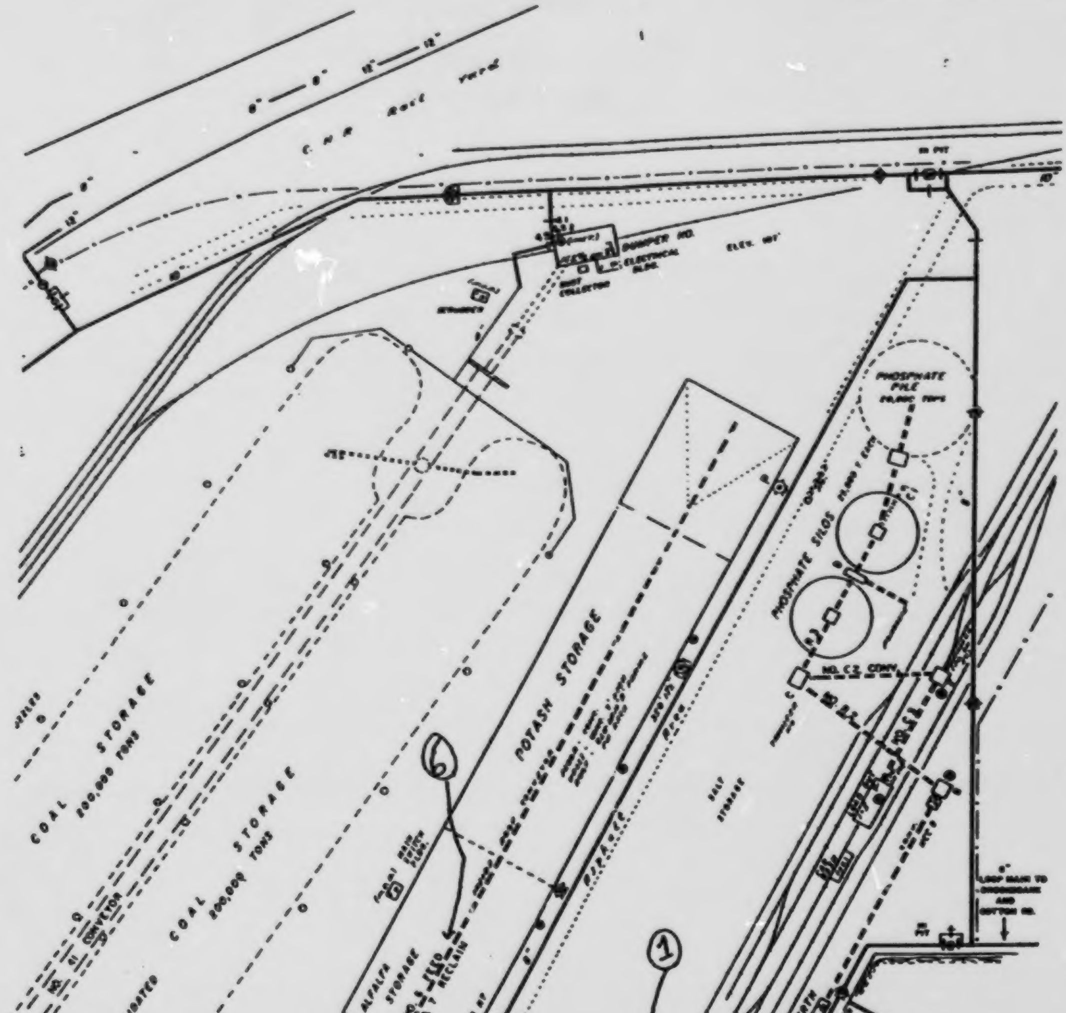
TECHNICAL DISCUSSION

Handling, Storage and Reclaim

Product received at Neptune Terminal (Figure 1) is unloaded in the unloading pit onto Conveyor #1 and transferred to Conveyor #3 where it can be directed to the warehouse (Conveyor #6) or to the vessel loading system (Conveyor #7). The following locations were identified as being potentially hazardous to product quality:

1. A 70 foot vertical drop off Conveyor #6 tripper into the warehouse.
2. A 70 foot vertical drop off Conveyor #3 onto Conveyor #7.
3. A 40 foot vertical drop off Conveyor #8 into the surge bin.
4. A 40 to 70 foot drop off Conveyor #11 and 14 into the vessel. This drop will vary depending if the belt flinger is used.

Figure 1


C N R Rail Yard
DUMPER HO.
ELECTRICAL BLDG.
DUST COLLECTOR
ELEV. 101'
PHOSPHATE PILE
PHOSPHATE SILOS
NO. C2 CONV.
COAL STORAGE
200,000 TONS
COAL STORAGE
200,000 TONS
LUSCAR CONSOLIDATED COAL LTD (ALBERTA)
CONVEYOR
POTASH STORAGE
ALFALFA STORAGE
NO. 7 RECLAIM
SALT STORAGE
NO. 3 CONV.
PHOSPHATE ROCK RECEIVING BERTH
LOOP MAIN TO GROUNDMAN AND COTTON RD.
6
1
3
7
8
9
12
NO. 8 CONV.
SURGE BIN
POLLUTION CONTROL
PARTS STORE
MOBILE EQUIP.
GASOLINE
DIESEL
POTASH BERTH
SHOP GARAGE
DOCK
NO. 12
BULK CARRIER
65,000 D.W.T. RANGE
MV STAR BALLARAT
Seaboard Dock
Burrard Inlet

Recommendations on how to eliminate these drops were made (Appendix III) and are seriously being considered by Canpotex, the Saskatchewan potash producer's marketing agency.

Although of less concern than the drops, product segregation and storage conditions in the warehouse were also investigated. No equipment is presently in place to reduce segregation and the installation of such equipment would result in significant expenditure. It was recommended that anti-segregation equipment be seriously considered for any additional expansions.

The only documented procedure used at Neptune Bulk Terminals (Canada Ltd.) is the "Dry Bulk Wash-Down Procedure" (Appendix IV). Serious considerations should be given to developing inspection procedures, plans and checklists for the unloading, storage and reclaim operations. Operators should also be provided with training in areas such as pile reclaim.

Sampling of Individual Railcars

Under certain circumstances individual railcars may be sampled as they discharge into the unloading pit. At the present time samples are taken manually with a small sample cup. Although the sampling frequency and sample size may be adequate for the chemical analyses which are performed they are not considered adequate for accurately determining the particle size distribution of the product.

Sampling equipment was designed and fabricated (Appendix V) by Kalium for the explicit purpose of obtaining representative samples from railcars. The manual sampler requires two operators, one on each side of the railcar as shown in Figure 2. The railcars hopper gate is opened approximately 6 to 8 inches and sampling starts as soon as a constant flow is obtained. The operators must cut the full stream without overfilling the sampler. After each pass across the stream the sampler is pulled back to its starting point and emptied into a bucket which is conveniently located next to the operators. After emptying the sampler the sampling operation is continued until the railcar is empty. The operators should maintain a constant sampling speed and frequency for each hopper being sampled. The sampling procedure has been included in Appendix VI.

The composite sample which is collected is then reduced to a 4-6 pound sample using the portable sample splitter (Figure 3) which was also designed and fabricated by Kalium (Appendix VII). The sample splitter consists of a Carpco Model No. 55-24-13X (Appendix VIII) which has been retrofitted with a larger hopper and a stand. The portable sample splitter can be easily assembled and operated by one operator.

Sampling of Incoming Material

Sampling of product received at Neptune Terminal is performed with an automatic sampling system at the discharge of Conveyor #3. The primary sampler (Figure 4) consists of seven probes which are mounted on a cylinder with a screw conveyor that transfers the primary sample to the secondary sampler. The secondary sampler consists of a Vezin divider (Figure 5) with a single sample collection slot. The following problems were identified with the basic design of the automatic sampling system:

1. Primary sampler does not take a full cross section of the stream.

2. Primary sampler is taking a larger sample from top of the belt due to its trajectory.

3. Primary sampler overfills.

4. Primary sampler is mechanically unreliable.

5. The screw which conveys product from the primary to the secondary sampler could be grinding the product.

6. The secondary sampler (Vezin divider) is taking too few increments and to small a sample.

FIGURE 2

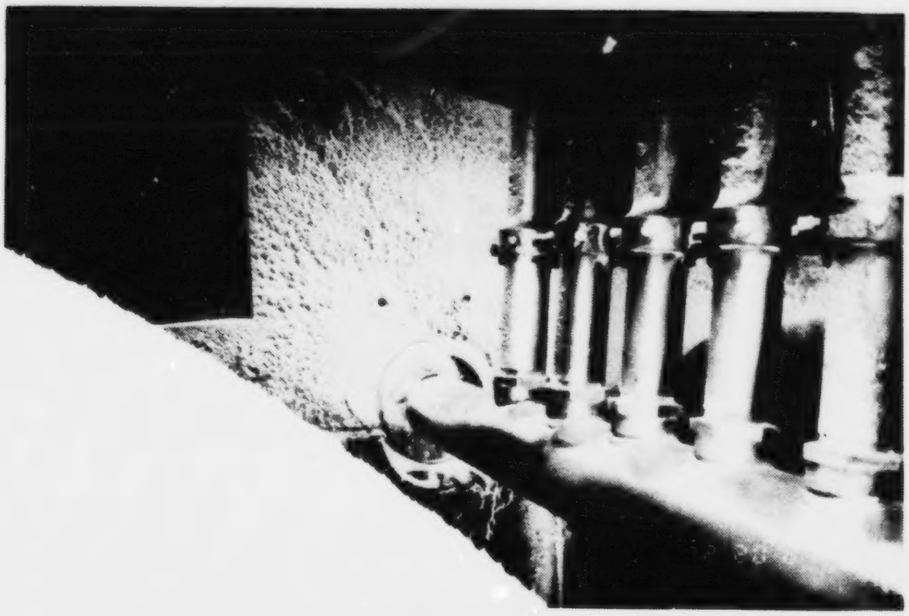
SAMPLING OF RAILCAR

FIGURE 3

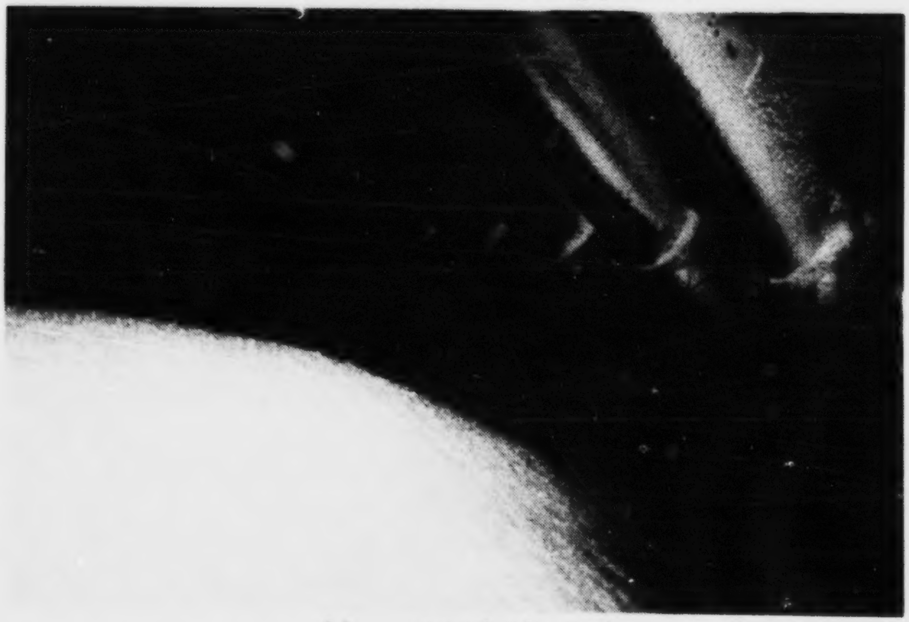
SPLITTING OF SAMPLES WITH MODIFIED GATED RIFFLE

FIGURE 4

PRIMARY SAMPLER AT DISCHARGE OF CONVEYOR #3

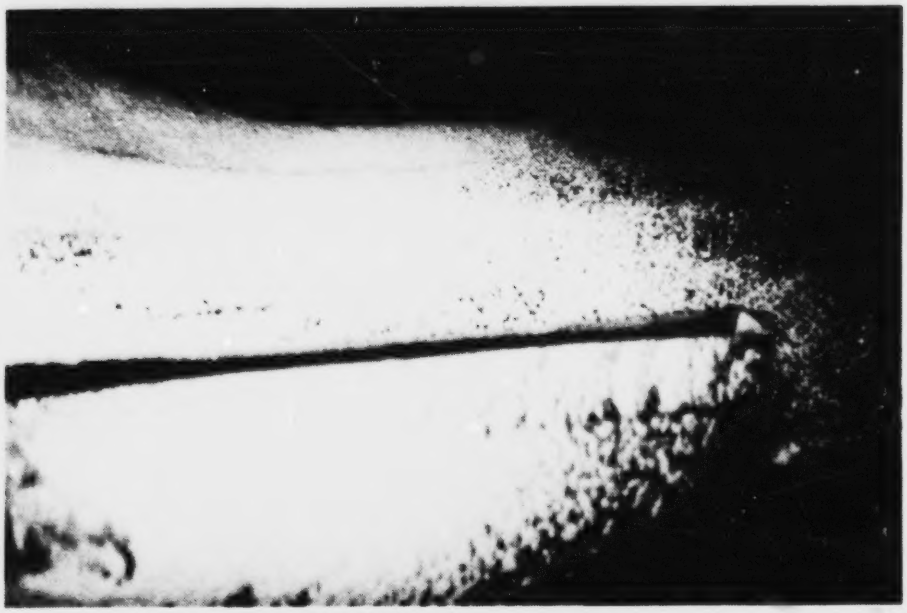


a) sampler in resting position



b) sampler in motion

FIGURE 4 (CONT'D)

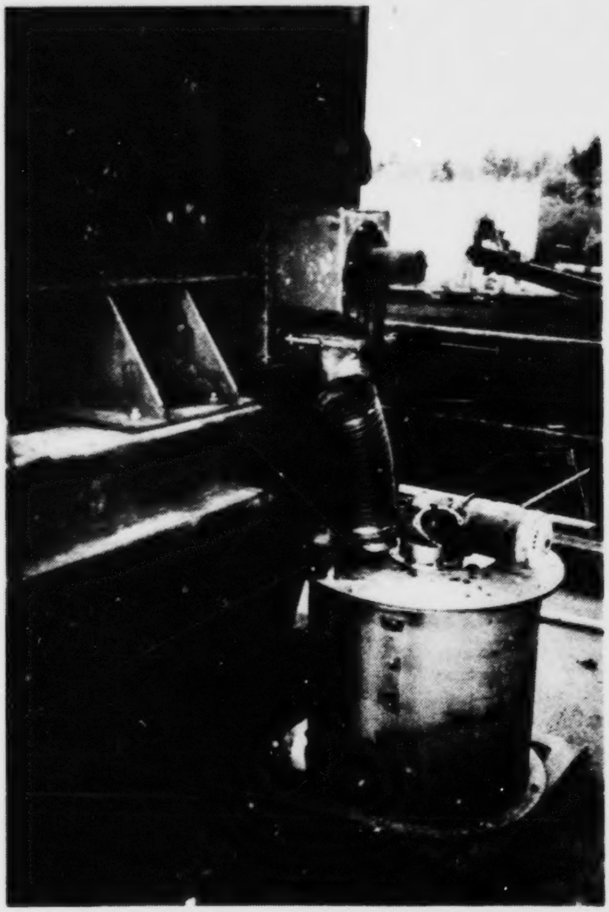


c) samples entering potash stream

d) sampler fully immersed in stream

FIGURE 5

SECONDARY SAMPLER (VEZIN DIVIDER) NEAR CONVEYOR #3



These observations are in agreement with previous findings by J. W. Mercks, Matrix Consultant Ltd (Appendix IX). There have always been concerns about the representativeness of the samples being taken by the automatic sampling system. The installation of a new sampling system had previously been considered but determined to be to expensive. Kalium has developed an automatic sampler which would be more economical and reliable (Appendix X).

Before any changes are considered or made to the automatic sampling system it was decided that the performance of the present sampling system should be determined. Samples collected at the minesite for unit trains would be compared to samples taken at Neptune. A survey (Appendix XI) of the sampling systems and procedures used by the minesites were performed to choose the plants which would have the best sampling system. The system used at Kalium (Table 1) was found to be the most thorough and was used for initial comparison.

Due to mechanical problems with the automatic sampler and the inability to repair due to heavy shipping only one shipment of Kalium Coarse grade potash was compared. The results (Table 2) indicated a significant discrepancy between the analysis performed by Kalium and Pkb Scania on the sample collected at Neptune.

Two potential causes for this discrepancy were identified:

1. The sample was damaged during transportation from Vancouver, B.C. to the minesite.

2. The size of the screens being used by Pkb Scania are incorrect or damaged.

To determine which one of these was causing the problem the following experiment was conducted.

Using a gated splitter a sample of Kalium Coarse was divided into 16 samples of approximately 100 to 150g each and three sets of five samples each were randomly chosen. The remaining sample was rejected. The first set was analyzed at Kalium using the same screen

TABLE 1

RESULTS OF SURVEY ON SAMPLING SYSTEM USED BY SASKATCHEWAN POTASH PRODUCERS ON EXPORT SHIPMENTS

	QUESTIONS	CCP	COMINCO	IMC K-1	IMC K-2	KALIUM	PCS-ALLAN	PCS - CORY	PCS - LANIGAN	PCS - ROCANVILLE
1	Export Products ① Sampled and Analyzed	1,17,10,11,18	1,4,7,18	2,7,17,18	2,4,6,9,13,17	3,5,13	1,13,16,18	1,4,10,12	1,10	2,4,8,18
2	Automatic or Manual Sampling System	Manual	Manual	Automatic	Manual	Automatic	Manual	Manual	Automatic	Automatic
3	Description of Sampling System	Stream Cutter with 3/4" opening and 12" long. Followed by 16:1 splitter.	Stream Cutter with 3/8" opening for Standard and 1/2" for Granular and Coarse	A 2" diameter pipe with a 12 mm x 460 mm slot rotates into product stream.	Stream Cutter with 1" opening	Dual Denver Stream Sampler with 3/8" opening followed by two 16:1 splitters.	Streamcutter with 1/2" opening	Streamcutter with 1 1/8" wide by 5 3/8" long opening.	Pelican type sample which cuts stream in an arc.	Modified Denver Slurry Automatic Sampler with 1/2" opening.
4	Sampling Frequency (# of Increments/ Railcar)	8	6	10	8	200-300	6 - 8	12 - 16	10 - 12	12 - 15
5	Size of Increment (kg)	1.9	1.2 - 1.4	0.5	0.3-0.4	1.3-1.9	0.9	0.5	0.5	0.2
6	Size of Primary Sample (kg) (Railcar Composite)	15.1	7.2 - 8.4	5.0	2.4-3.2	380	5.5 - 7.3	5.5 - 7.3	4.5 - 6.8	2.3
7	Size of Final Sample (kg) (Laboratory Samples)	0.7	0.5	0.6	0.45 - 0.60	1.5	1.9	1	0.5	0.9
8	Is each railcar sampled ?	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
9	Sample reduction device used in Laboratory	Standard Riffle	Standard Riffle	Standard Riffle	Standard Riffle	Gated Riffle	Rotary Sample Divider	Rotary Sample Divider	Standard Riffle	Standard Riffle
10	Analyses performed on each railcar ② sampled	13		1,13	1,2,3,7,13	13	13 on 4 Railcar Composite			1,13
11	Analyses performed ③ on three composites	1,2,9,12,13	1,10,11,13 ④ (SGN)	1,10,11,13,	1,2,3,7,9,13 ⑤	1,2,3,4,5,6,7,10,13	1,2,13,(10 and 11) ⑥ ⑦	1,2,7,8,10,11,13,14 ⑧	1,2,8,10,12,14 ⑨	1,2,3,7,8,10,11,12,13,14 ⑩

1,2,3 Key is in survey form in Appendix XI.

4 Wet degradation test performed on Granular.

5 Ca, Br and Amine analyses performed on Industrial White Soluble.

6 12 car composite.

7 1 railcar only

8 Degradation test Granular 210

9 Degradation test on Granular 210

10 Degradation test on Granular and Coarse

Form: RJAG/03/28/88 TAB.1

TABLE 2

PARTICLE SIZE ANALYSIS RESULTS

FOR KALIUM COARSE

LOADED ON TOKYO RAINBOW ON DECEMBER 19, 1987

Sample Description	Kalium Shipment Unit Train Average	PKB Composite Sample	
Laboratory Analyzed By:	Kalium Lab	Pkb Lab	Kalium Lab
Tyler Mesh	(Cumulative Weight Percent Retained)		
8	17.1	17.55	11.4
10	51.6	----	44.2
14	89.1	91.70	87.4
20	97.1	----	96.0
28	---	99.45	---
35	99.5	----	99.0

(sieve) series. The second set was carefully packaged and sent via courier to Pkb Scania for analysis. The third set was carefully packaged, sent via courier to Pkb Scania and immediately returned to Kalium for analysis. This was done to evaluate the effect of transportation on a carefully packaged sample. The results (Table 3) indicate that there is no statistical difference between the samples. The initial discrepancy (Table 2) was, therefore, due to the sample being damaged in transportation.

Since only one set of data was obtained we were unable to statistically determine if there is any bias between sampling systems. Additional data will be collected to complete the evaluation. However, if mechanical problems with the primary sampler at Neptune persist, serious consideration will have to be given to designing and/or purchasing a more reliable primary sampler.

Sampling of Outgoing Material

Although an automatic primary sampler is in place on top of the surge bin at the discharge of Conveyor #8, it is not operational and has been cannibalized to keep the primary sampler at the discharge of Conveyor #3 operational. Samples for outgoing material are taken from Conveyor #3 when directly loading from railcars to a vessel and from Conveyor #7 when loading from the warehouse. A small sample cup is used to obtain samples of product on the belt and a 20 to 30 pound sample is usually taken.

An automatic sampling system which will take representative samples should be installed for outgoing Products. The ideal location would be after the material has gone through the surge bin, such at the discharge of Conveyor #9 and #12 but it is probably not feasible to place an automatic sampling system at these locations. If samples are taken after the surge bin product degradation and/or contamination can be more easily detected. The top of the surge bin is probably the only possible location.

Table 3

COMPARISON OF KALIUM AND PKB SCANIA PARTICLE SIZE DISTRIBUTION ANALYSIS

Tyler Mesh	Measurement	Sample Set #		
		1	2	3
.+8	Mean	21.94	22.04	22.06
	Std. Dev.	0.53	0.87	0.38
	Variance	0.28	0.76	0.15
.+14	Mean	96.94	97.72	97.23
	Std. Dev.	0.36	0.13	0.39
	Variance	0.13	0.02	0.15
.+28	Mean	99.60	99.78	99.58
	Std. Dev.	0.16	0.08	0.30
	Variance	0.03	0.01	0.09
.-28	Mean	0.40	0.22	0.42
	Std. Dev.	0.16	0.08	0.33
	Variance	0.03	0.01	0.11

Sample Set #1 Analyzed by Kalium
#2 Analyzed by PKB Scania
#3 Analyzed by Kalium after being shipped to Vancouver and immediately returned to Regina via courier.

Sampling at Offshore Destinations

Canpotex requested that at least two shipments of Granular grade potash be monitored in 1988 or 1989 during transport, unloading and storage to an offshore destination (Australian or Brazil) in order to determine the impact of the handling systems and environmental conditions on product quality.

Obtaining a representative sample of products at offshore locations is very difficult because of the difference in the unloading systems at each offshore destination. Detailed information (drawings, pictures) of the transfer points is required to determine if the required sampling can be achieved. Equipment for manually sampling potash streams and reducing large samples was designed and built by Kalium. The equipment is essentially identical to the equipment which was designed for sampling railcars (Appendix V & VII) except that minor modifications were made to the sampler so as to allow sampling from different positions.

The timely physical and chemical analysis of the samples which are taken could also prove difficult. This is especially true with a compacted product such as Granular potash which can drastically change in physical strength from day to day due to changes in internal moisture. It was, therefore, decided that the required analyses should be performed at the sampling location. The equipment required to perform the analyses will vary depending on the equipment available at the various sampling locations. In the next phase of this project the analytical procedures and equipment will be more clearly defined.

Sample Preparation

The Pkb Scania laboratory will typically receive a 20 to 30 pound sample from manual or composite sampling for each lot received or shipped. A mixing-by-coning procedure (Appendix XI) followed by incremental division with a Jones riffle divider is used to reduce the sample size. The sample was reduced to approximately 2,000 g and from this sample exactly 100 g would be poured out for particle size distribution analysis.

Part of the remaining portion of the material would be used for chemical analysis.

In the mining industry coning is viewed as a good method for mixing materials. In the fertilizer industry coning is known to cause significant segregation (demixing) of the particles in blending grade potash. It was recommended that they perform their sample reduction with a gated riffle (Appendix XIII) which is recognized as being one of the best riffle dividers.

The pouring of a 100 g portion out of the bag for particle size distribution analysis will increase the variability of the analyses due to segregation of the particles when they were initially poured in and as they are being poured out. It was recommended that the Jones riffle (and eventually the gated riffle) be used to reduce the sample down to an increment of approximately 100 - 150g.

Analysis

In most cases Pkb Scania uses Standard AOAC procedures to perform the analyses required. These procedures are acceptable but may require slight modifications or can be simplified when analyzing muriate of potash. A committee which consisted of one representative from the quality assurance department of each minesite was formed to review the analytical procedures being used by Pkb Scania.

The committee decided that AOAC procedures were acceptable but recommended that a sample exchange between the minesites and Pkb Scania be initiated. The samples would be analyzed by each participant using both AOAC procedures and the procedures typically used by each minesite. The analysis results will be analyzed by the committee and, if required, improvements made.

One of the important objectives of the task force in the future will be to develop procedures for the sampling, sample preparation and analysis of various potash grades being received at Neptune and Vancouver Wharves, or shipped to offshore destinations. These procedures would be similar to the coal sampling procedure (Appendix XIV) which was supplied by Pkb Scania.

TASK TWO: DEVELOPMENT OF NEW MEASURES OF PRODUCT QUALITY.

DESTRUCTIVE TESTING

In the first phase of this study PCS developed a degradation test which was adopted by the industry as a standard degradation test. PCS had also developed a dedusting test (Appendix V) and in this second phase of the study industry members adopted the PCS dedusting test as a standard. A dedusting apparatus was fabricated for each participating minesite and for the Pkb Scania laboratory. Round Robin testing will be performed in the third phase of this project.

New measures of product quality which can improve upon the present measurements must continue to be developed if potash producers and Canpotex want to determine and/or improve product quality.

Moisture Absorption

International Mineral & Chemical Corporation Limited (IMC) developed a humidity and temperature control chamber and performed preliminary work to determine the amount and rate of moisture absorption of their potash products (Appendix XVI).

Their testwork indicated that the higher the relative humidity (RH) the greater the amount of moisture which was absorbed. The lower purity potash grades (<62% K_20) absorbed a small amount of moisture at 20 and 50% RH and leveled off after four hours or less. At 60% and higher RH they continued to absorb moisture even after 24 hours. The high purity (>62% K_20) potash grade only continued to absorb moisture over the 24 hour period when the RH was 90%. The difference in moisture absorption between the lower purity and high purity potash grades can be attributed to the lowering of the critical humidity of the product due to high levels of NaCl, $MgCl_2$ and $CaCl_2$ in the lower purity potash grades.

The effect a mixture of anticaking/antidusting agents (3% Armeen-T in #1000 oil) would have on product moisture absorption was also investigated by IMC (Appendix XVII). The product was treated with 1.9

kg/t and exposed to 70% relative humidity for 24 hours. The anticaking/antidusting mixture had either no effect or slightly increased the amount of moisture absorbed by the product.

Serious consideration should be given to developing an economical and reliable humidity and temperature control cabinet which could be used in conjunction with the other standard product quality test which have been developed. This could lead to a better understanding of the effects moisture absorption has on product strength, dusting and caking characteristics. Further work should also be performed to determine if any anticaking and/or antidusting agents have moisture repelling qualities.

Acoustic Emission

A report (Appendix XVIII) that the USDA Agricultural Research Services had developed a method for rating the hardness of a single kernel of grain was investigated. The measurement is accomplished by passing a single kernel of grain through a small grinder and measuring the acoustic emission from the grinding process. The inventors (David R. Massie and Karl H. Norris) state that "hard grade grinds slightly louder than soft ones." When contacted at the USDA's Federal Grain Inspection Service in Kansas City the inventors stated that changes in moisture content effect the acoustic emission and that they had not found a solution to this problem at this time. Although communication will be maintained with the USDA future work in this area will concentrate on locating and using a consultant who specializes in the field of acoustic emission testing.

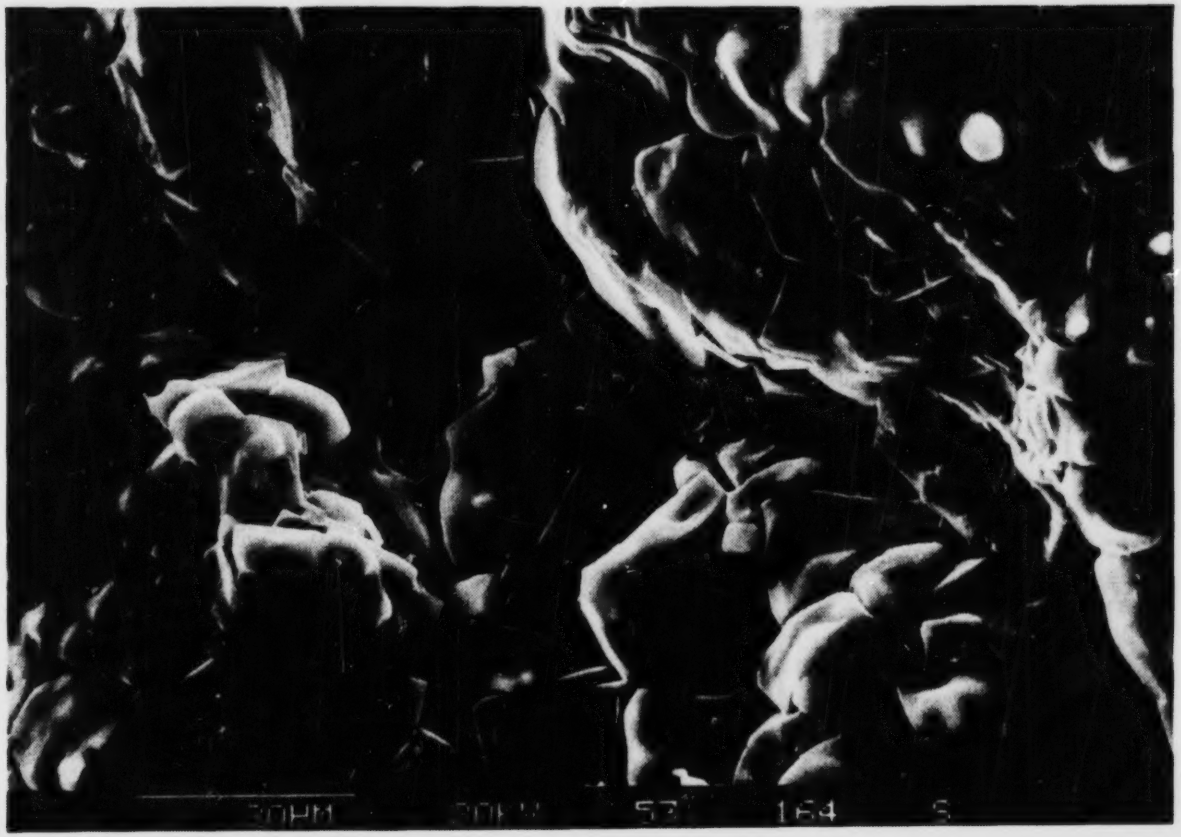
NON-DESTRUCTIVE TESTING

Non-destructive testing of compacted potash flake and/or product which would provide fast and accurate measurements of quality would allow potash producers to more easily monitor and improve the quality of potash grades containing compacted material.

Experienced potash workers can visually determine the quality of potash flake produced in a compaction circuit. For producers of red potash a light red color usually signifies a poor quality flake while a dark red color

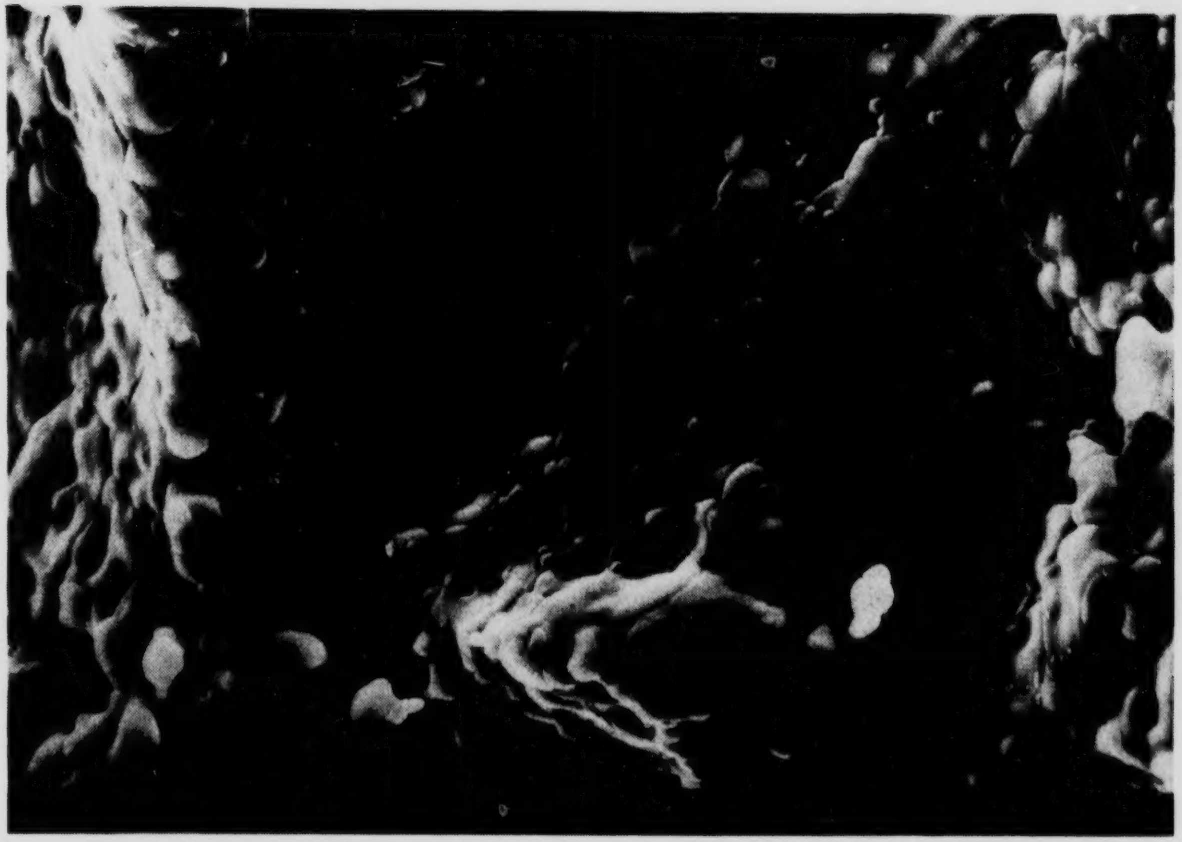
FIGURE 6

SCANNING ELECTRON MICROSCOPY PICTURES OF A COMPACTED
POTASH SURFACE MAGNIFIED 500X


30UM
30KV
531
164
S

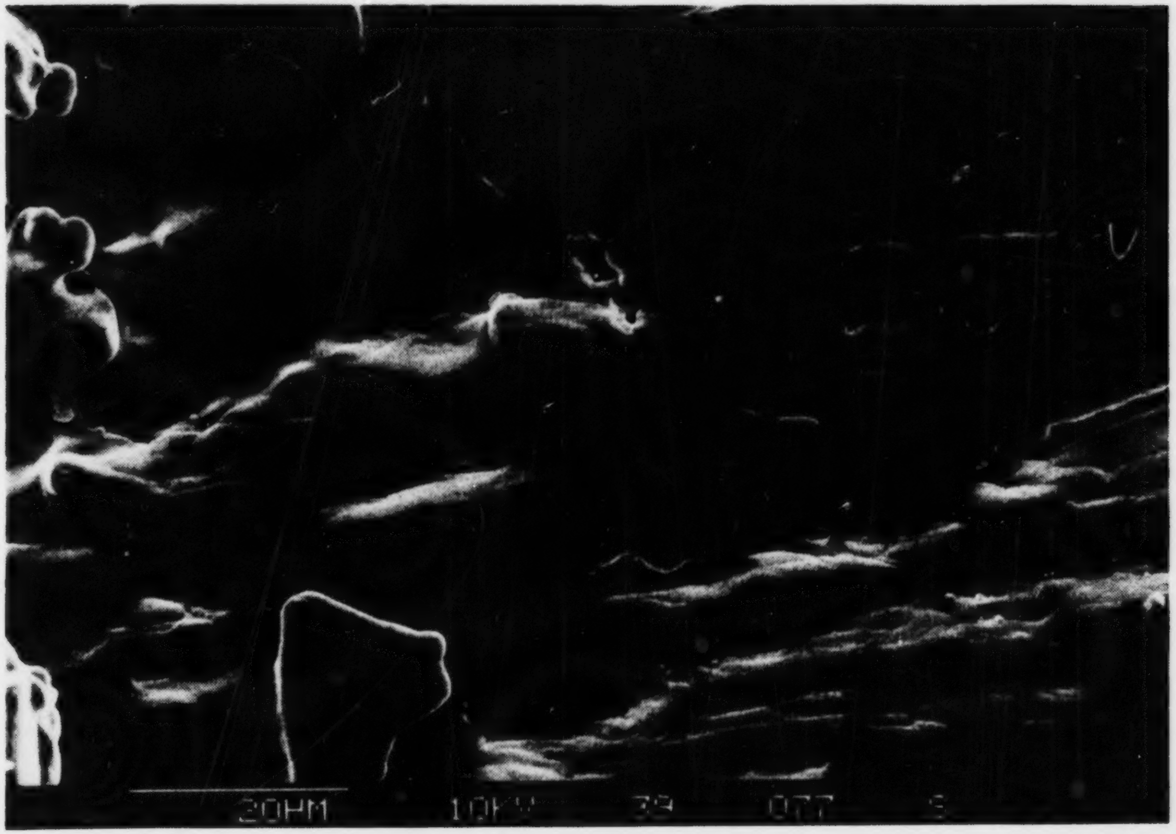
a) very loosely packed potash particles

FIGURE 6 (CONT'D)



b) potash particles are tightly packed but boundaries are still present

FIGURE 6 (CONT'D)


20µM
10KV
39
OTT
S

c) potash particles are very tightly packed to the point where boundaries are no longer evident.

usually signifies a good quality flake. For producers of white potash a white flake signifies poor quality while an off-white, opaque and/or glassy flake signifies good quality. In both cases the sound emitted by the flake when impacted upon also changes with the quality of the flake.

The differences in the appearance of potash flake have been attributed to the elimination of void spaces between potash crystals. The scanning electron microscope pictures in Figure 6 provide a visual explanation.

The following methods of performing non-destructive testing are being considered:

- impact echo
- light reflectance
- color
- gas permeability
- acoustic emission

In this report only the first two possibilities have been investigated.

Impact Echo

A very crude experiment was conducted to determine if good and poor quality potash flake emitted different sound patterns when impacted upon. The apparatus (Figure 7) consists of a spring loaded steel hammer (actually a safety eye glass lens tester), a crystal microphone, a Macintosh computer and the appropriate hardware and software to convert the microphone signal to a graphics display of sound waves. The results (Figure 8) indicate that there is a substantial difference between the sound patterns emitted by good and bad compactor flake. Further testing in this area will be considered.

Figure 7

Acoustic Analysis of Flake Samples

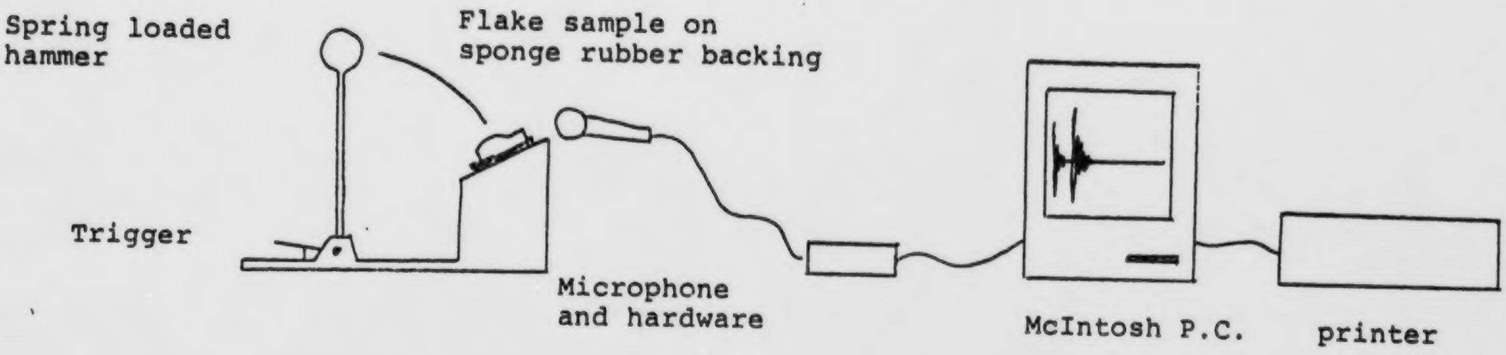
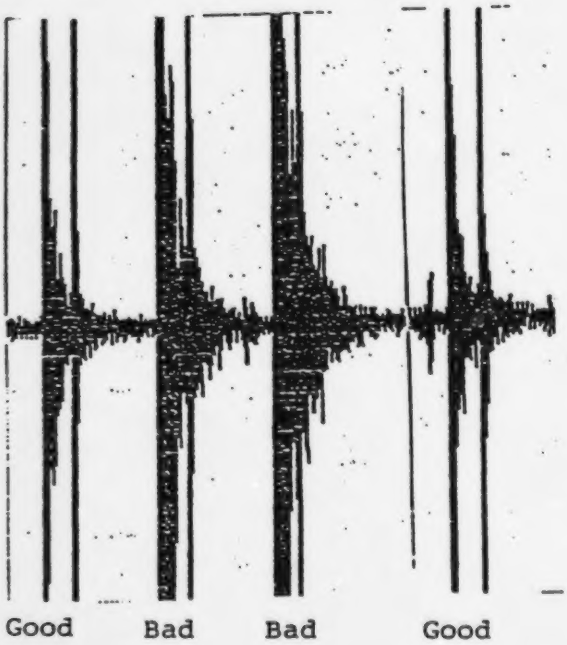

Spring loaded hammer
Flake sample on sponge rubber backing
Trigger
Microphone and hardware
McIntosh P.C.
printer

Figure 8

Sound decay waves emitted from "good" and "bad" compactor flake samples after being hit with a steel ball.



Light Reflectance

A Minolta Chroma Meter CR 200b (Appendix XVII) which is capable of providing highly accurate measurements of chromacity and illuminance was used to evaluate the difference in reflections between good and poor quality potash flake.

Synthetic pellets were produced using a Carver Laboratory press (model B) with 24,000 pound capacity. The pellets fabricated using a plunger type mold which made circular pellets with an area of one square inch. Approximately 7 g of product was used for each pellet. A mold releasing agent (Miller-Stephenson, M5-122) was used to ensure easy removal of the pellets. Good quality pellets were fabricated by pressing the product for 15 seconds with 24,000 psi of pressure. Poor quality pellets were fabricated by pressing the product for 15 seconds with 5,000 psi of pressure. Samples of Kalium (white), Cominco (red) and Lanigan (reddest) compactor feed were evaluated and a mixture of Kalium/Cominco (pink, 90/10) compactor feed was also evaluated.

The percent reflectance readings were taken from the feed material and pellets. Measuring of the reflected light from the compactor feed was performed by first overfilling a watch glass with compactor feed. A glass slide was then slid across the top of the product while resting on the watch glass. The Chroma Meter was then placed on the glass slide and a reading taken. Measuring of the reflected light from the pellets was performed by holding the Chroma Meter perpendicular to the flat surface. In each case the poor pellets reflected more light than the good pellets (Table 4).

The effect of surface imperfections on reflected light readings was investigated. A good pellet produced from Kalium compactor feed was lightly scratched with sandpaper and then gouged with a nail. Ten consecutive readings were taken and the results (Table 5) indicated that

Table 4

Synthetic Pellets and Compactor Feed Material

Values in % Reflectance

Sample Description		Feed*	Poor** Pellet	Good** Pellet
White (Kalium)	Mean	81.0	86.2	74.0
	Std. Dev.		0.9	1.7
Pink (Kalium/ Cominco)	Mean	76.3	77.8	64.4
	Std. Dev.		0.6	3.3
Red (Cominco)	Mean	35.9	35.9	22.8
	Std. Dev.		0.8	2.0
Reddest (Lanigan)	Mean	32.7	31.6	18.0
	Std. Dev.		1.8	0.9

*Single reading
** Average of 10 readings

Table 5

Effect of Scratches on Reflected Light Readings

Pellet Description	Mean % Reflected Light	Standard Deviation
Smooth	74.00	1.71
Light Scratches	72.74	1.99
Light Scratches and Gouges	71.76	1.72

the roughness of the surface appears to have a small affect on the amount of light reflected.

The effect of compacted potash flake shape on reflected light reading was investigated. Flake produced on an Allis Chalmers compactor with corrugated rolls was used. The amount of light reflected from the peaks and valleys of the flake was determined by taking 10 readings at each location. The following results were obtained:

Table 6

Effect of Flake Shape on Reflected Light Reading

	% Mean Light	Standard Deviation
Peak	66.78	1.5
Valley	63.48	1.9
Random	65.48	2.4

The results indicate that the Chroma Meter measuring head can be positioned in either or both the peaks and valleys with only a small impact on the results. It is important to note that the measuring head was at 90° to the flake sample. Changes in the measuring head to sample angle did drastically affect readings.

A two feet by one foot flake sample produced by a Koppern compactor with pocketed rolls was used to determine if there was a change in the amount of light reflected from flake produced in the middle to the edge of the roll. Material from the edge of the roll is typically less compacted than material from other locations. Results (Table 7) of the readings from the centre to the edge of the flake clearly indicate that the flake quality progressively worsens towards the edge of the roll.

The amount of light reflected by potash flake appears to have good potential as a non-destructive test. Further testwork will be conducted in this area.

Table 7

% Reflected Light Readings from Fresh Koppern Flake

Location	Sample	% Reflect	Mean	Std. Dev.
Edge of Roll	1	86.8		
	2	88.2		
	3	86.5		
	4	86.1		
	5	82.5	86.0	2.1
4 to 5" from edge of roll	6	83.4		
	7	82.9		
	8	83.2		
	9	84.1		
	10	82.8	83.3	0.5
8 to 10" from edge of roll	11	81.0		
	12	81.0		
	13	81.6		
	14	82.5		
	15	81.7	81.6	0.6
12 to 15" from edge of roll	16	81.1		
	17	81.0		
	18	81.3		
	19	81.9		
	20	80.1	81.3	0.5
16 to 20" from edge of roll	21	80.0		
	22	77.9		
	23	77.0		
	24	78.5		
Middle of Roll	25	78.9	78.5	1.1

CONCLUSIONS & RECOMMENDATIONS

From the audit of the offshore quality assurance program it is evident that significant improvements could be made to ensure that Canpotex is supplied with quality products and in turn able to supply offshore customers with quality products. The quality assurance deficiencies which were identified should be addressed, especially the areas of product sampling and handling. The development of new measures of product quality which will assist in establishing the storage and handling characteristics of various products during or after the manufacturing stage should be continued.

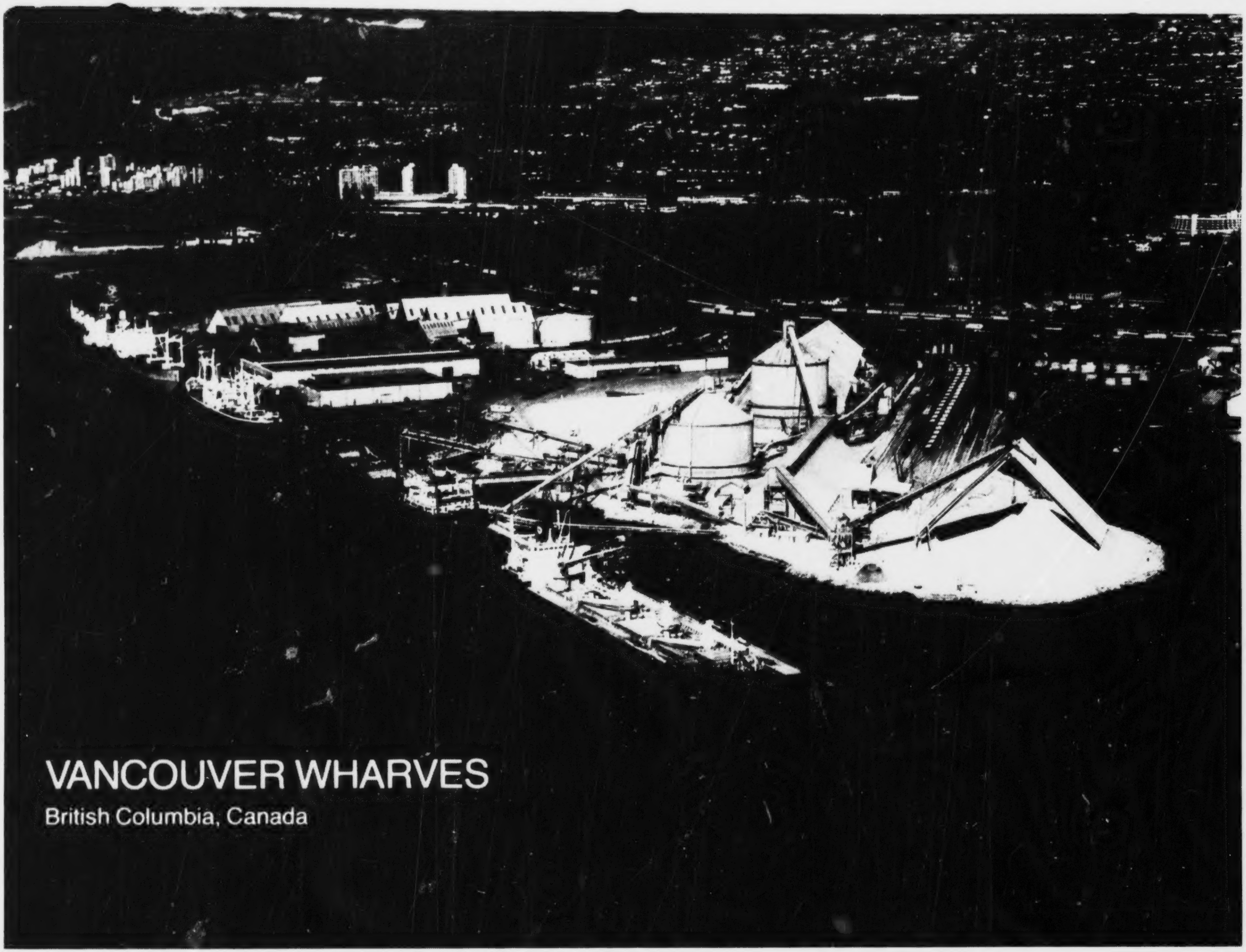
Canpotex and the potash producers must continue to work together to improve the quality assurance program. Canpotex should give serious considerations to adding to their staff a technical person with experience in quality assurance. This person could work with the potash companies and offshore customers to develop and maintain the quality assurance program.

APPENDIX I

BROCHURE ON NEPTUNE BULK TERMINAL LTD

APPENDIX II

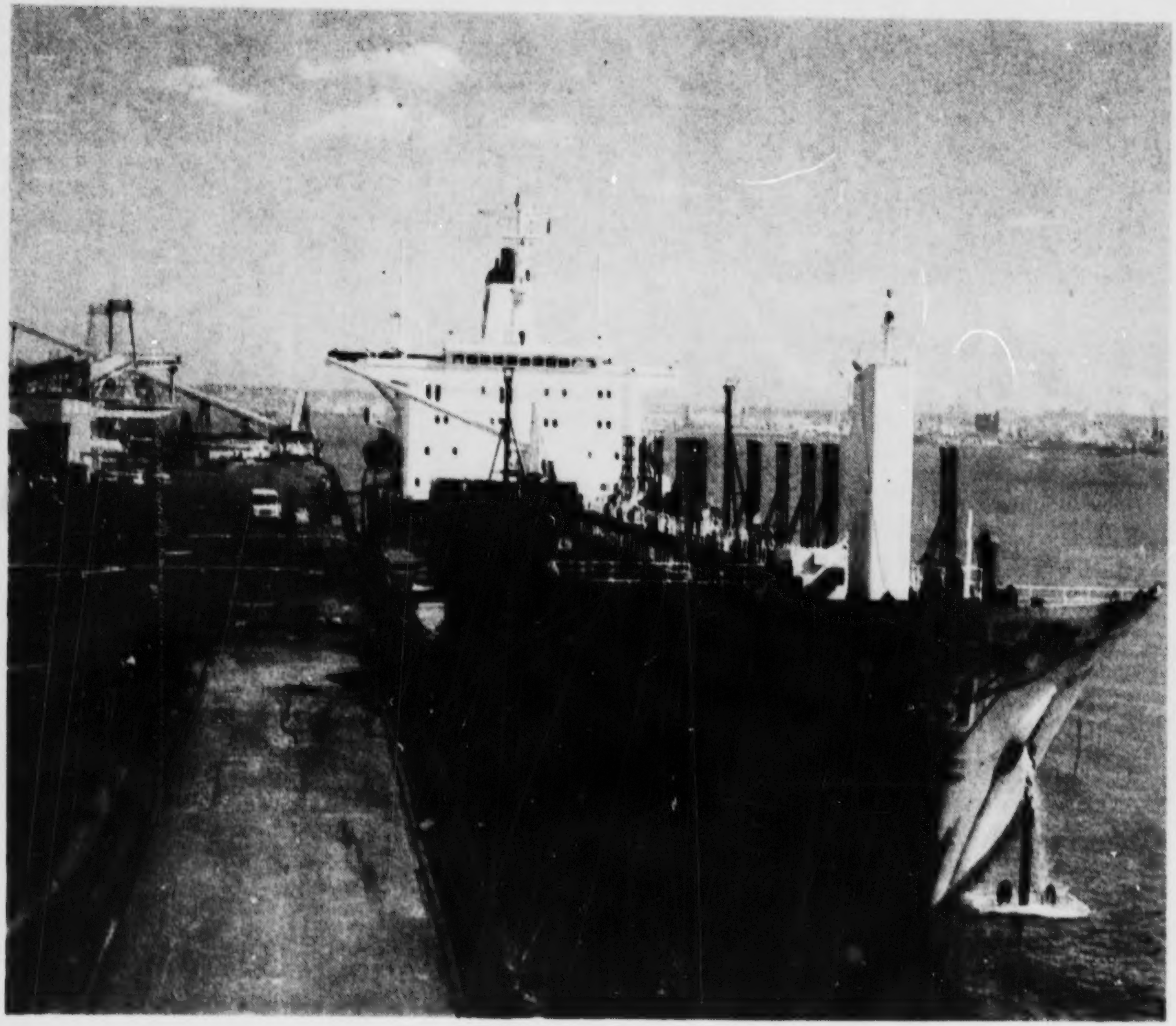
BROCHURES ON VANCOUVER WHARVES



VANCOUVER WHARVES

British Columbia, Canada

VANCOUVER WHARVES LTD.
A Subsidiary of Williams Hudson Canada Limited



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Company Officials

D.V.L. ODHAMS
Chairman & Chief Executive Officer

H.R. WOODS
President and General Manager

J. MACKAY
Executive Vice-President

B.E. THIRSK
Financial Director

R.J. SPRAY
Sales Manager

R.P. DUEY
Traffic Manager

Bankers:

Canadian Imperial Bank of Commerce
Royal Bank of Canada

Auditors:

Peat, Marwick, Mitchell & Co.
Vancouver, B.C.

Legal Advisors:

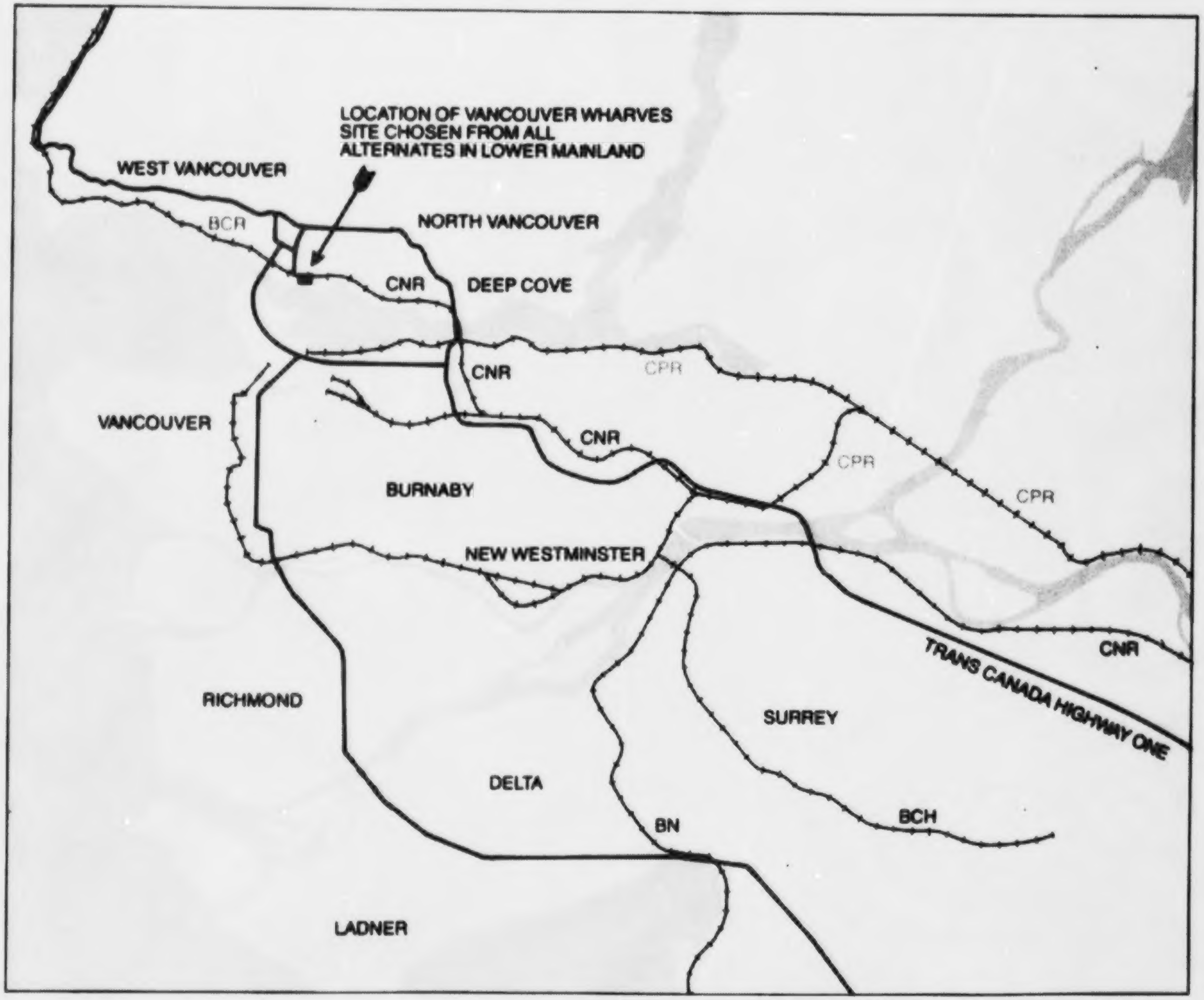
Campney & Murphy,
Vancouver, B.C.

Insurance Brokers:

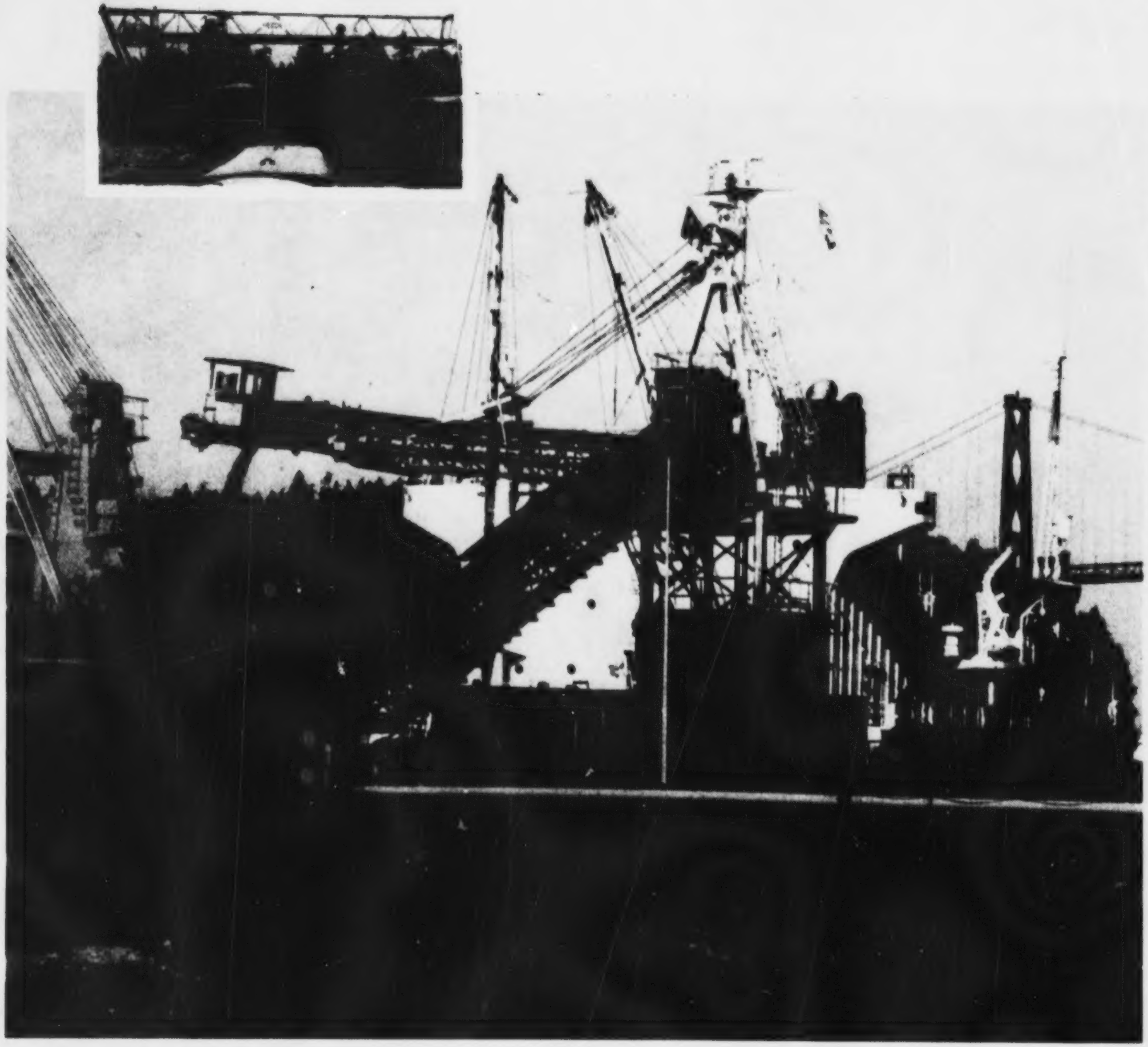
Tomenson Saunders Whitehead Limited

THE BACKGROUND TO VANCOUVER WHARVES LTD.

In the period 1958 to 1960 the growth of the mining industry in British Columbia and Alberta demonstrated a need for at least one, and possibly two, bulk loading terminals. The Williams Hudson Group who had been associated with the dock and stevedoring business since the last century carefully examined the harbour of Vancouver. The criteria was that all railway systems should be capable of quoting to and reaching the terminals, that it have easy access to the main trans-Canada highway, that it be within easy reach of the centre of Vancouver, that it have deep water, excellent load bearing qualities and that it be capable of expansion. All these advantages were provided by the final site chosen and in 1959 the ground clearing and initial dredging was begun. Up to the end of 1978 the terminal has handled over 50,000,000 tonnes of cargo, mainly for export, of which over 32,000,000 tonnes have been sulphur and potash. The terminal handles over 120,000 rail car movements a year and serves exporting mines which are located as far as 3200 km from the coast. The terminal employs some 200 men and women and has contributed over 5,000,000 dollars per annum in payroll and development taxes etc. to the National, Provincial, and Municipal economies. In its development and growth this private enterprise terminal has paid its own way and continues to do so to the benefit of the nation's economy.


LOCATION OF VANCOUVER WHARVES SITE CHOSEN FROM ALL ALTERNATES IN LOWER MAINLAND
WEST VANCOUVER
BCR
NORTH VANCOUVER
CNR
DEEP COVE
CNR
CPR
VANCOUVER
CNR
CPR
CPR
BURNABY
NEW WESTMINSTER
CNR
TRANS CANADA HIGHWAY ONE
RICHMOND
SURREY
DELTA
BN
BCH
LADNER

THE CONCENTRATE DIVISION
No. 1 Berth



The unprecedented development of base metal mining in Western Canada necessitated expanded concentrate facilities at dockside. Present storage facilities have a capacity for 240,000 tonnes. The system is capable of an annual throughput of 2,000,000 tonnes per year. A variety of transportation systems, including rail, truck and containers, deliver concentrates to the dock facility. The annual volume to date is in the order of 1,000,000 tonnes.

Recent advances include the relocation of the Gradall rail car unloader, a new triple-spur rail track for faster railcar handling, and individual, weather-proofed shed conveying systems. A Heede straddle crane removes and replaces car covers.

Reclaiming from covered stockpiles is achieved by front-end loaders with a bucket size of 12 tonnes capacity. The ship loader system, which is supplied by a controlled flow from the plate feeders in the reclaim hoppers deliver concentrate to bulk carrier at an average production rate over the loading period of 600 tonnes per hour. The vessel remains stationary while the loader travels the length of the vessel delivering to hatches by means of its loading boom.

THE LIQUID DIVISION

No. 1 & No. 2 Berths

At present there are three tanks designated for the storage of methanol. Two of the tanks have a capacity of 3,785 cubic metres and the third is of 15,142 cubic metres capacity. There is an underground 25.4 cm receiving line parallel to the methanol rail road track with 8-10.16 cm suction hoses spaced 19.76 meters apart. The loading line is 25.4 cm and runs underground to Berths 1 and 2 where it feeds a 20.32 cm flexible loading hose. The system is flexible in that it is capable of receiving and shipping either:

1. From tank cars or trucks to barge or ship or:
2. From ship or barge to tank cars or trucks.

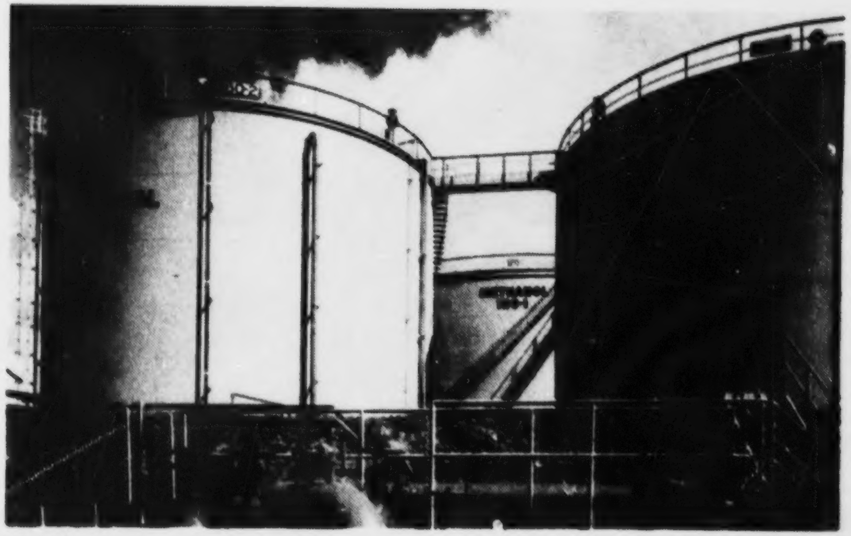
The facilities can handle liquids directly or through storage tanks and they can also transfer from one tank to another.

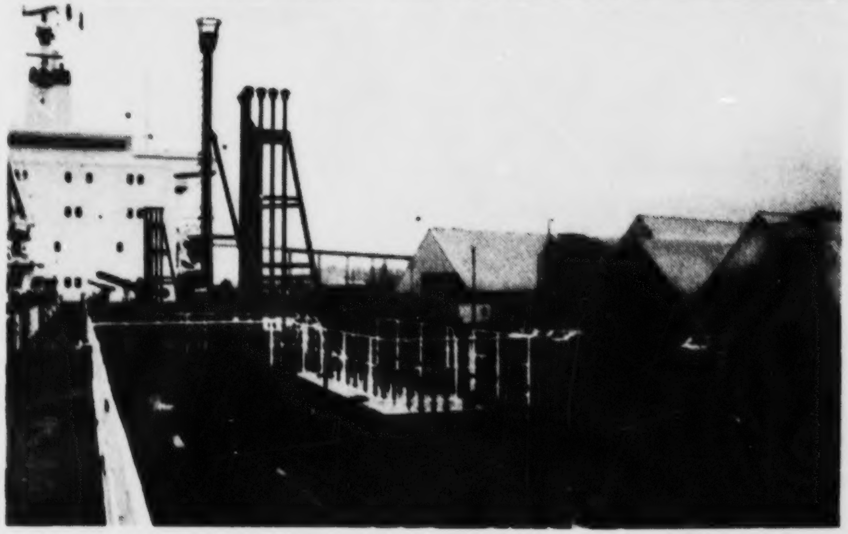
Each of the three tanks is equipped with the following:

1. Fire extinguishing foam system.
2. Dry air system which maintains (one ounce p.s.i.) air pressure at tank top.
3. Refrigerator system for emission control of methanol vapours.
4. Automatic level readout gauge and temperature gauge at bottom of tank.
5. Flame arrestor vents and pressure relief valves on top of tanks.
6. Inter connecting walkways from tank to tank.
7. One 20.32 cm over head filling line, one 25.4 cm suction line, one 15.24 cm suction stripping line and one 5.08 cm tank clean out line. Six fire hydrants are installed and three of these have monitors (fixed play pipes) which are manually operated. On the East side of the railroad methanol track there is a sprinkler type water deluge system.

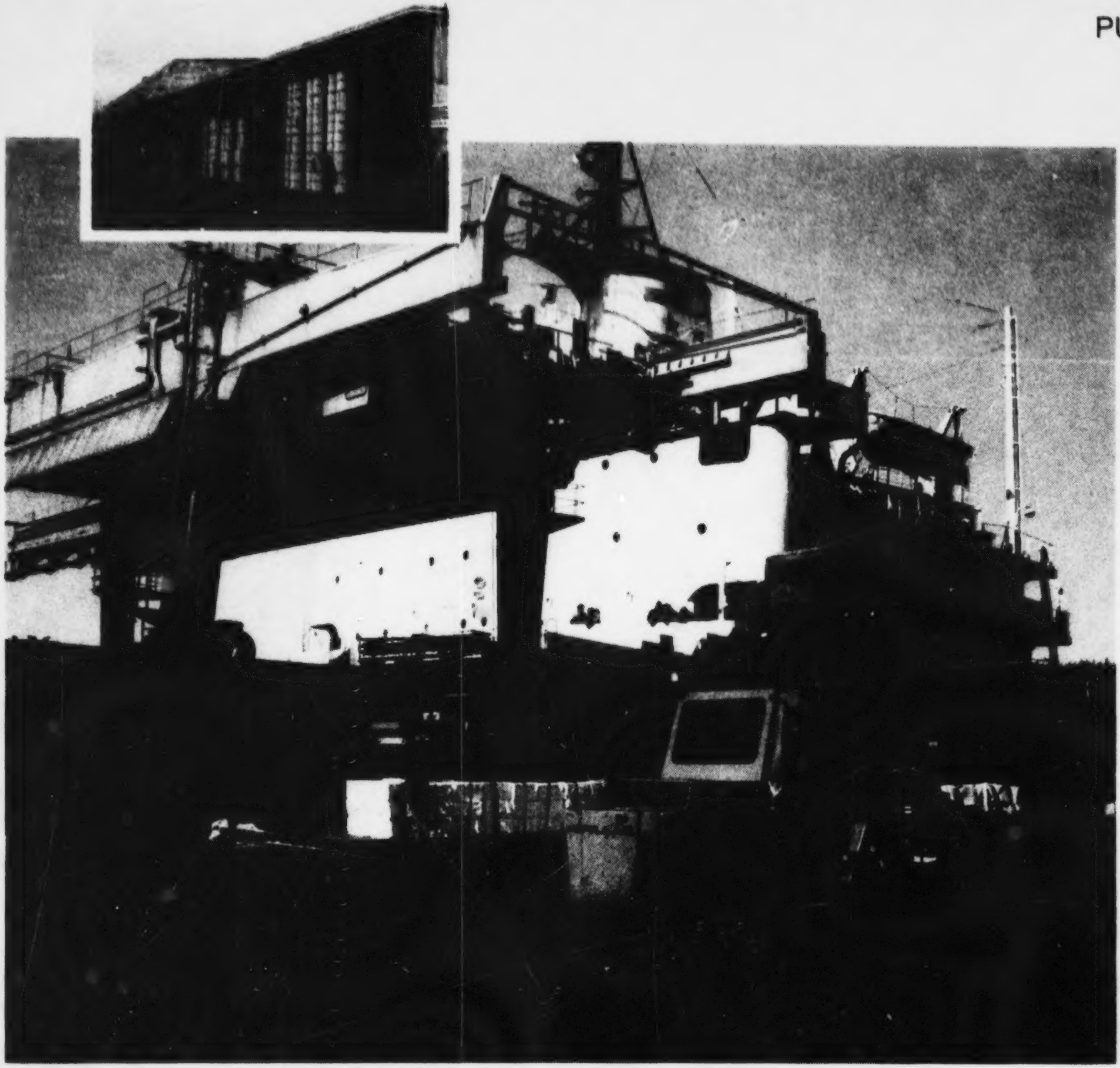
There is also a stainless steel underground pipe system for loading direct from rail cars to vessel which is capable of taking such liquids as acetic anhydride.

Storage facilities can be expanded as needed to meet shippers requirements.





PULP AND PAPER DIVISION
No. 2 & No. 3 Berths



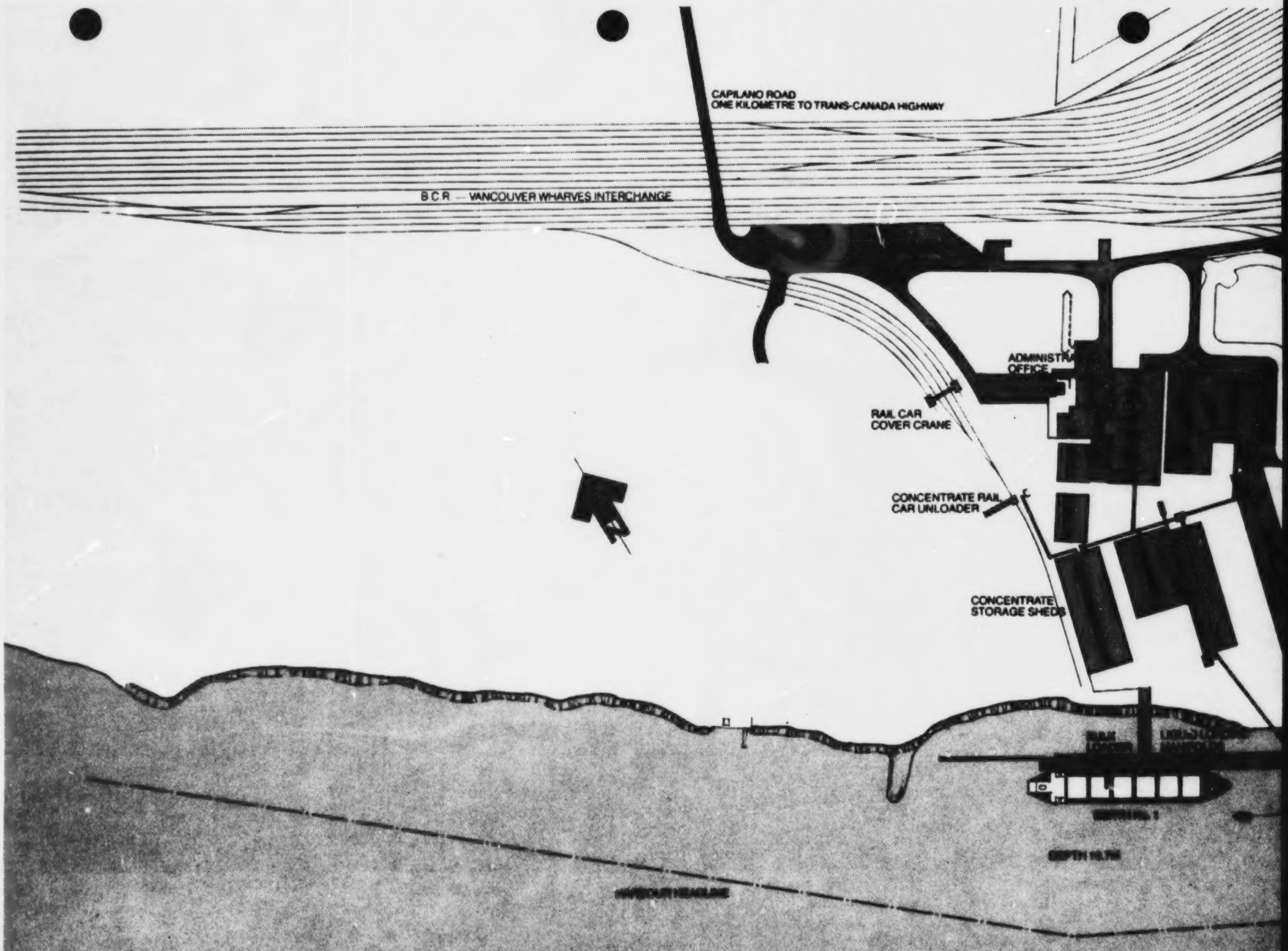
Adjacent to Berths 2 and 3 there are four fully sprinklered all weather warehouses for the receiving, storing, and shipping of pulp and paper products.

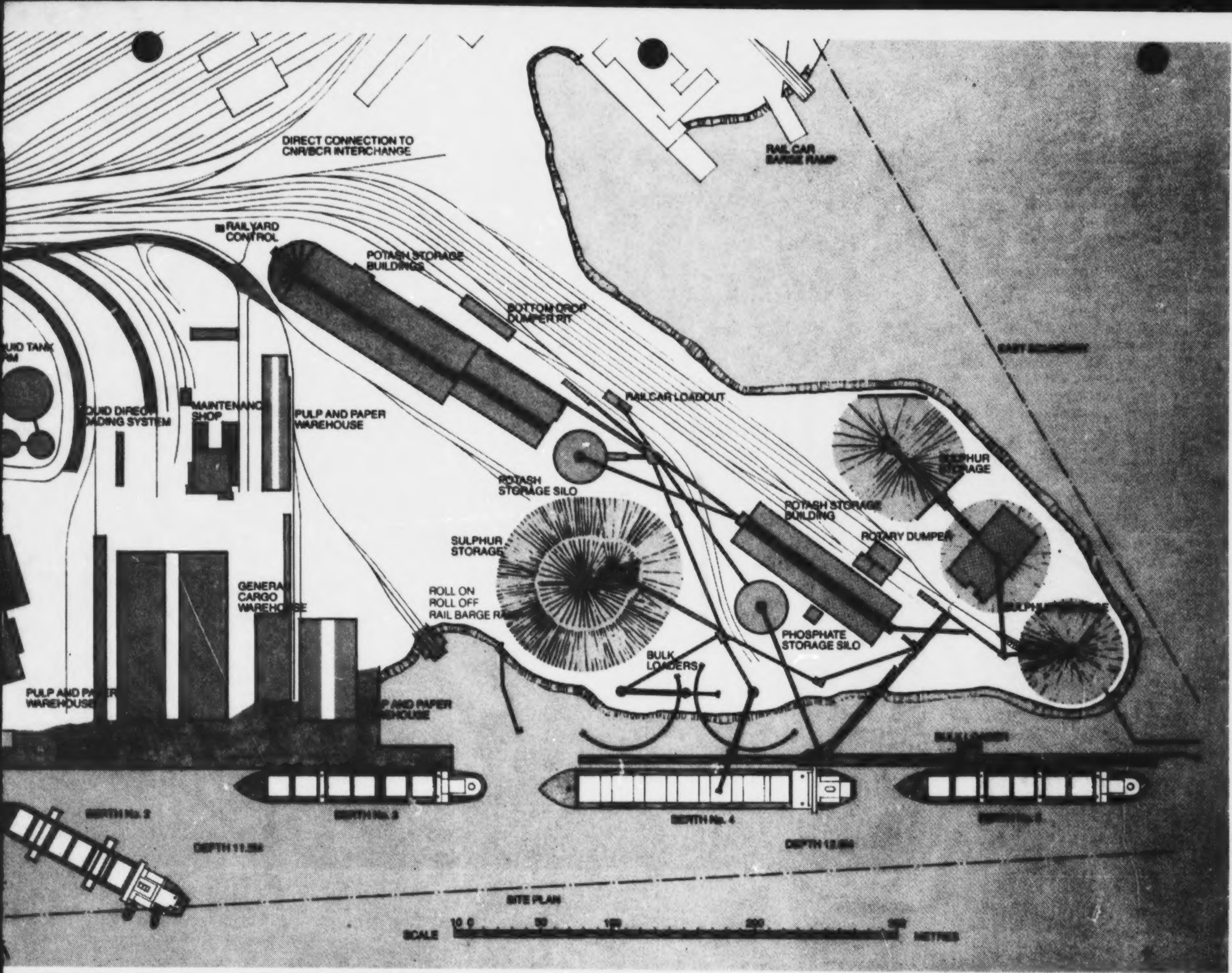
These sheds have a combined floor space of 14,628 square meters.

The receiving system is such that the terminal can receive from all modes for all its customers at the same time.

The unloading ramps can accommodate sixteen rail cars at once for unloading, apart from trucks when required. Clamp trucks are the standard mobile equipment for handling pulp and paper.

Vancouver Wharves Ltd. has been one of the developers of new and efficient pulp and paper handling techniques and has further developed the terminal expertise for export pulp from the interior mills of Western Canada. It remains in the forefront of the development of pulp handling techniques.

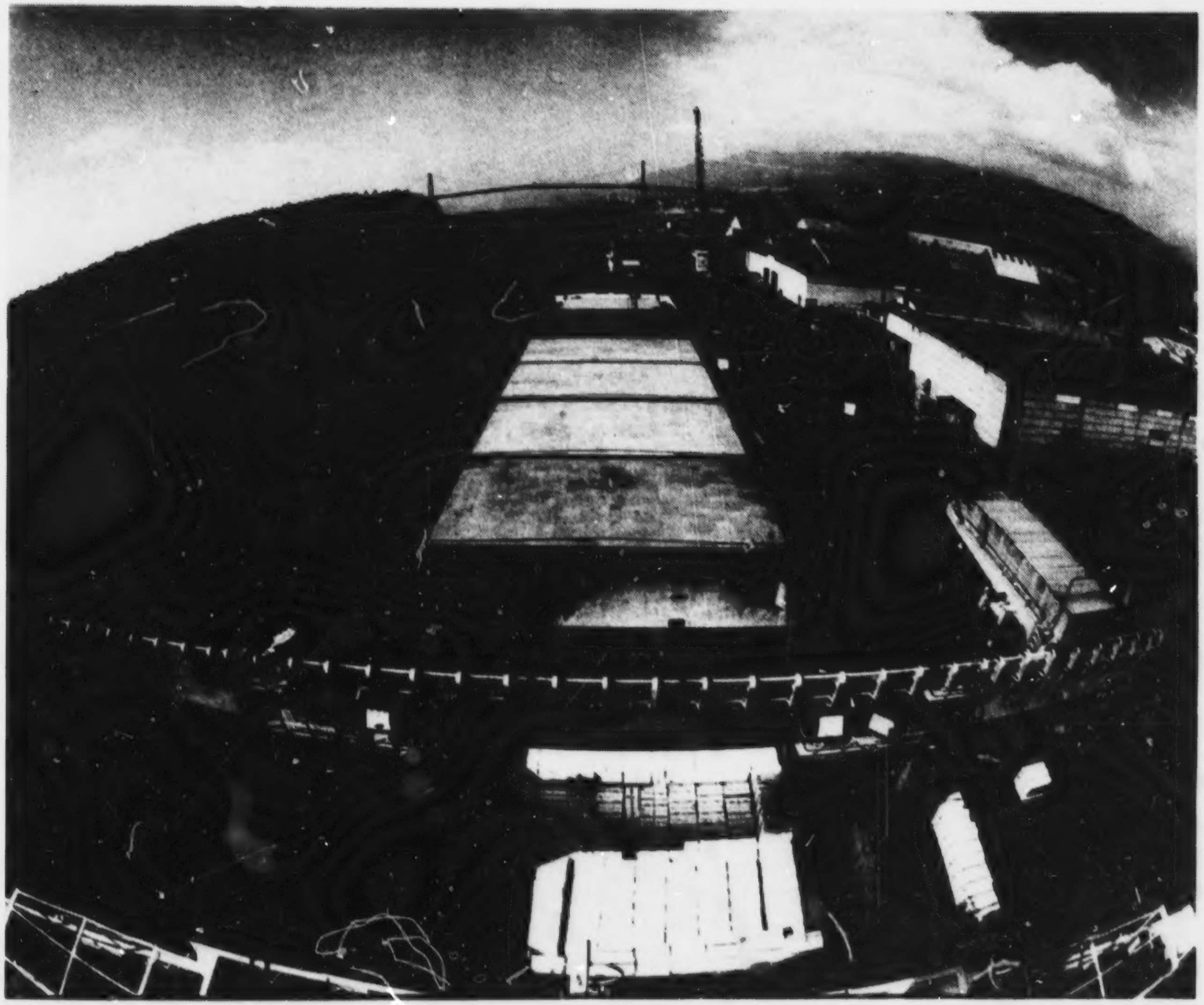

CAPILANO ROAD
ONE KILOMETRE TO TRANS-CANADA HIGHWAY
B C R — VANCOUVER WHARVES INTERCHANGE
ADMINISTRA
OFFICE
RAIL CAR
COVER CRANE
CONCENTRATE RAIL
CAR UNLOADER
CONCENTRATE
STORAGE SHEDS


DIRECT CONNECTION TO CNR/BCR INTERCHANGE
RAIL CAR BARGE RAMP
EAST BOUNDARY
RAILYARD CONTROL
POTASH STORAGE BUILDINGS
BOTTOM DROP DUMPER PIT
RAILCAR LOADOUT
LIQUID TANK FARM
LIQUID DIRECT LOADING SYSTEM
MAINTENANCE SHOP
PULP AND PAPER WAREHOUSE
POTASH STORAGE SILO
SULPHUR STORAGE
POTASH STORAGE BUILDING
ROTARY DUMPER
SULPHUR STORAGE
SULPHUR STORAGE
GENERAL CARGO WAREHOUSE
ROLL ON ROLL OFF RAIL BARGE RAMP
PHOSPHATE STORAGE SILO
BULK LOADERS
PULP AND PAPER WAREHOUSE
PULP AND PAPER WAREHOUSE
BULK LOADER
BERTH No. 2
BERTH No. 3
BERTH No. 4
BERTH No. 5
DEPTH 11.2M
DEPTH 12.8M
SITE PLAN
SCALE
10 0
50
100
200
300
METRES

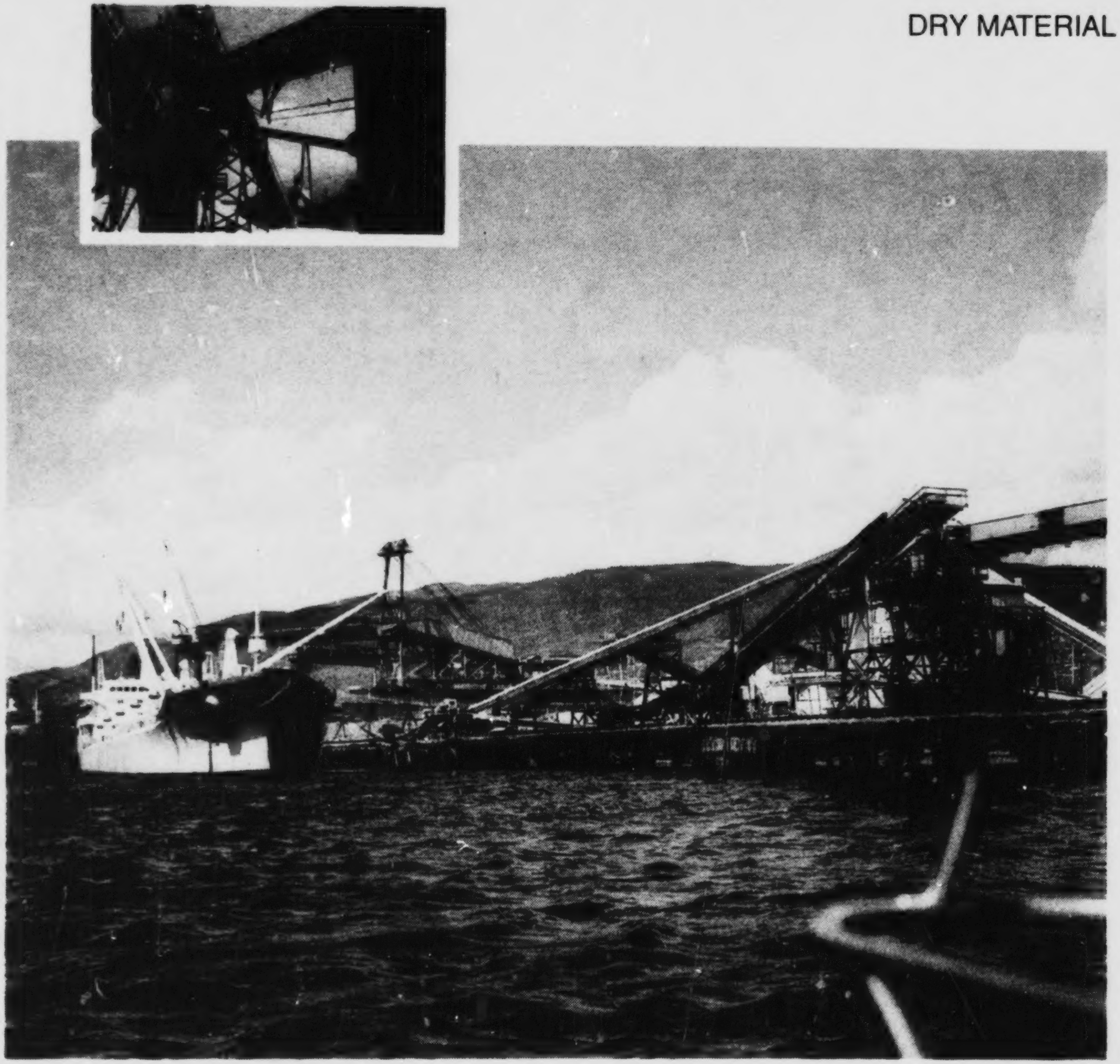
GENERAL CARGO DIVISION

Vancouver Wharves Ltd. has the physical and technical ability to be flexible in the types of cargo that it can handle. Up to 15 acres of outside storage is available for handling general cargo. In particular the terminal has handled large quantities of steel and pipe apart from automobiles and containers. The terminal handles bagged and drummed goods.

The roll-on roll-off rail barge slip offers many possibilities for handling general cargo in transit to and from the outlying ports.



DRY MATERIAL BULK LOADING DIVISION



This aspect of the terminal operations is designed to accommodate those import and export industries which are trading in large quantities of bulk material such as sulphur, potash, phosphate rock, fertilizers, animal feeds, grains etc. The rail cars are unloaded on to belt systems which take the material to covered storage in either shed or silo or to open storage when desirable. Direct loading to vessel is always available.

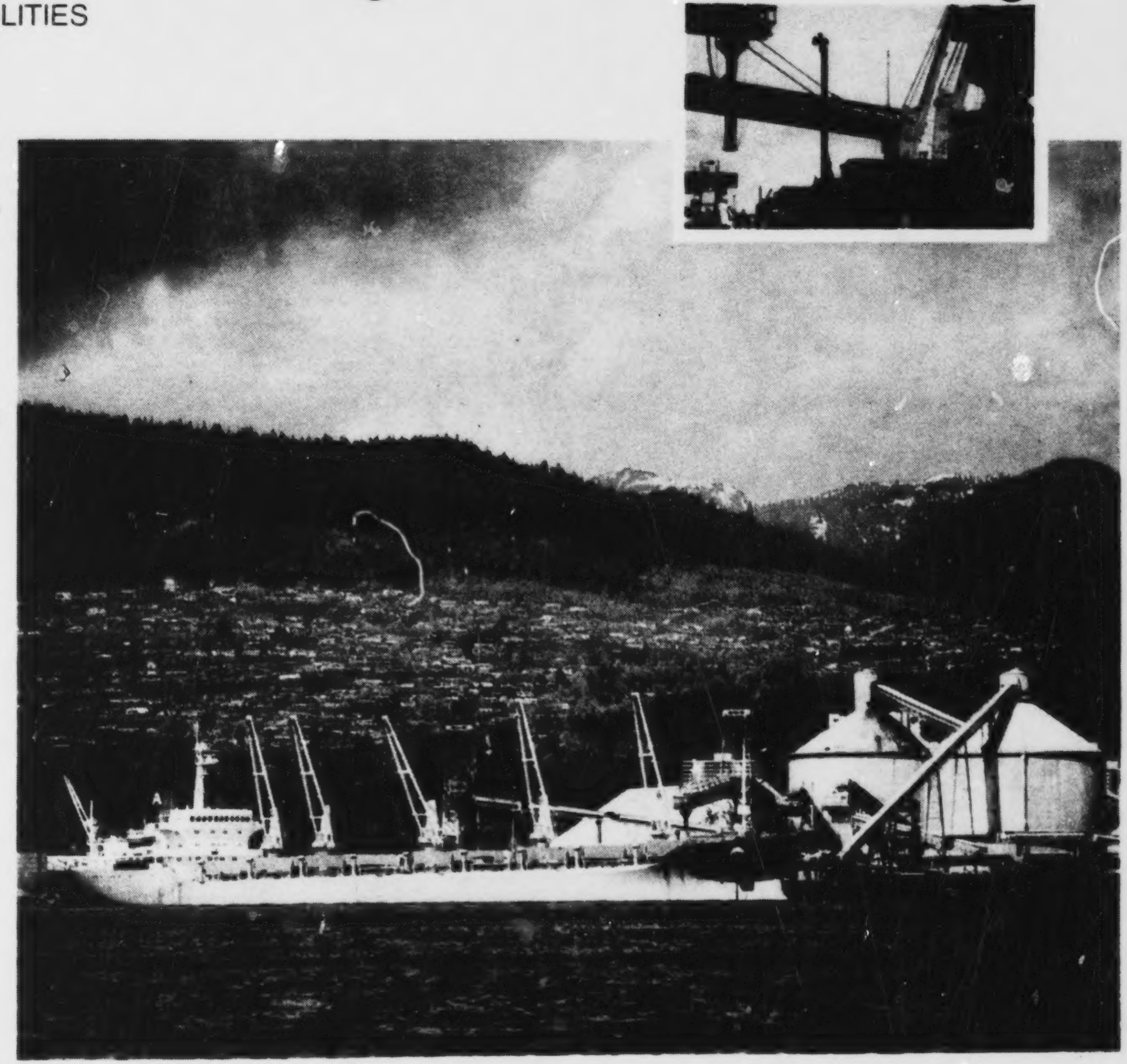
Both Berths No. 4 and No. 5 are independent and interchangeable allowing two vessels to load the same or different commodities simultaneously. No. 4 Berth also serves as a passive bulk discharging dock with hoppers capable of receiving bulk commodities from ship's gear at the rate of 1,000 t.p.h. This system feeds a 45,300 tonnes phosphate rock silo and a rail car load-out system.

POTASH HANDLING FACILITIES

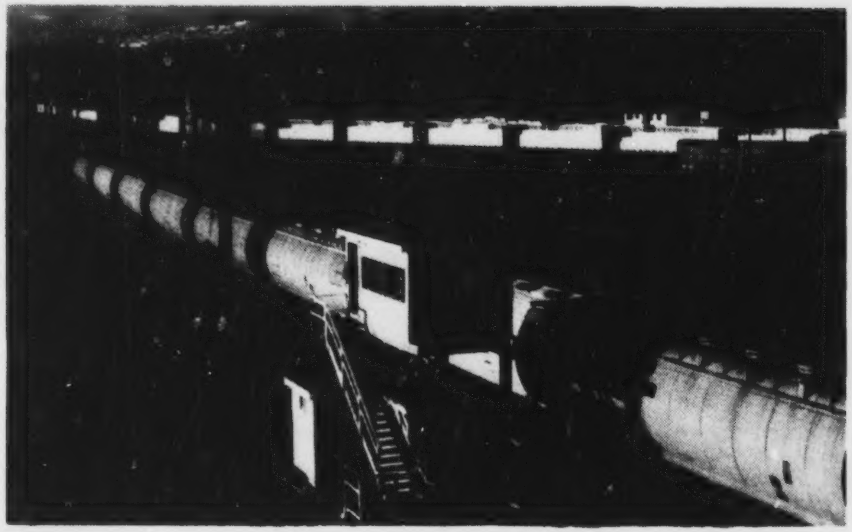
The potash facilities are designed to store 154,000 tonnes of potash of different grades. The storage is of three distinct types, the largest area being a flat storage area under shed cover with a side reclaiming system consisting of conveyor belts running alongside the Eastern supporting wall. These conveyors feed into the main belt system. The second type is again on a flat base under cover but with an underground reclaiming system. The last is the silo system which can contain up to 36,300 tonnes and reclaim is by gravity on to an underground belt system.

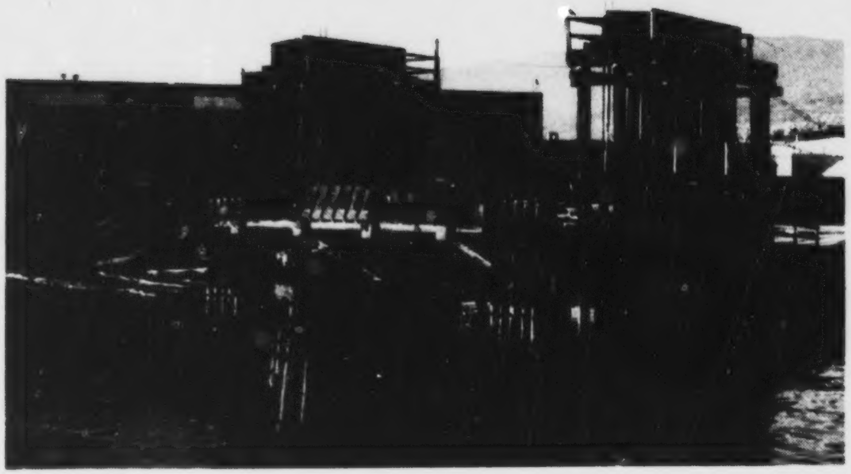
Rail cars, from unit trains, are bottom dumped in a specially designed unloading shed where operators can control the door openings from sunken walkways alongside the cars. From all potash storage areas material can be fed to the dry bulk loaders at berths No. 4 and No. 5.

Rates of loading at No. 4 berth can exceed 20,000 tonnes in 8 hours from live stockpile. Further expansion of these facilities is possible.

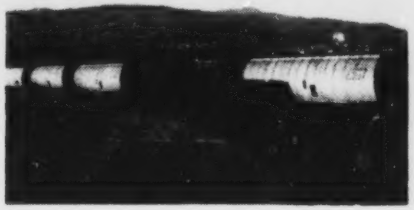


THE RAIL SYSTEM





Vancouver Wharves Ltd. operates and maintains a fully independent, integrated rail service utilizing its own rail track and motive power units.



With the complexity of operations at a large terminal, such as Vancouver Wharves Ltd., it is essential to have a well developed rail receiving interchange and distribution system. From the western end of the dock area the British Columbia Railway feeds into the track interchanges whilst from the eastern end, cars are delivered from the C.N.R. etc. by a special track that bypasses the B.C.R. main yard.

There is a total of 14 kilometres of track on the site all of 100 lb. rail. The system has two main marshalling areas and is capable of handling unit trains of potash and sulphur simultaneously with daily deliveries of concentrates, pulp, liquids, etc.

Spur lines include a three track concentrate working area, a two track liquids spur, three pulp and paper spurs, one spur used for car storage, one rail barge spur, and one maintenance spur for the six locomotives that are used in the system. The maintenance spur is not only equipped with locomotive pit and overhead gantry but has a sanding tower. The maintenance complex is situated on this spur.

The barge ramp receives and loads rail cars from and to rail barges on a three track spur.

Over 120,000 rail car movements in and out per year are effected here.

The Maintenance Department

The terminal carries out its own maintenance programmes on a round the clock basis. A staff of 9 and a crew of 40 is responsible for the maintenance of fixed plant and mobile equipment. Trades include electricians, millwrights, plumbers, welders, mechanics and carpenters.

SULPHUR HANDLING

Sulphur in its various forms is received at the terminal in unit trains of up to 105 cars each. The sulphur is unloaded from the cars by means of a rotary dumper after which the cars are automatically returned via a spring loaded switch system to the marshalling area.

Sulphur is stockpiled in four main storage areas which have a designed total capacity of 324,000 tonnes.

One storage area is covered by a large A frame which also acts as a support for the conveyor belts and chutes. From this area sulphur is recovered by means of underground conveyors. The other stockpile areas are serviced by inclined conveyors resting on supporting concrete towers. Reclaim is by front-end loader at all stockpiles.

Sulphur can be loaded from all three loaders at Berths 4 and 5 which can all be reached from all stockpile area.

Further expansions of these facilities is still possible.

TECHNICAL DATA

BERTH	1	2	3	4	5
MATERIAL	Wet Concentrate Liquids	Pulp & Paper Liquids	Pulp & Paper General Cargo	Dry Free Flow	Dry Free Flow
Length of Berth Face		486 m		402 m	
Total Length Dredged Length		566 m		482 m	
Overall Length to Dolphins		597 m		539 m	
Mooring dolphins beyond Wharf?	Yes	N/A	Yes	Yes	Yes
Fenders	Dolphins	Continuous Fender Piles	Continuous Fender Piles	Dolphins	Dolphins
Depth	10.7 m	11.3 m	11.3 m	12.8 m	12.8 m
Belt Width	0.76 m	N/A	N/A	1.37 m	*1.07 m
Belt Speed	213 m/min	N/A	N/A	243 m/min.	198 m/min.
Capacity Max	900 tonne/hr	N/A	N/A	3000 tonne/hr.	*1810 tonne/hr.
Capacity Net	Varies between 50% and 75% of maximum depending on ship & cargo flow characteristic				
Trimmer	By bulldozer	N/A	N/A	Belt Trimmer 900 tonne/hr. max.	Belt Trimmer 900 tonne/hr. max.
Maximum Outboard Reach of Shiploaders from Berth Faces	15 m	N/A	N/A	17.5 m to 29 m varies	18.2 m
Height of trimmer above low tide	15.5 m	N/A	N/A	23 m	21 m
Height of Wharf Deck above low tide	6.7 m	6.7 m	6.7 m	6.7 m	6.7 m
Loader type	Travelling	N/A	N/A	Twin Quadrant	Travelling
Travel	115 m	N/A	N/A	177 m	152 m

*Conversion of #5 Shiploader to 1.21 m belt, 243 m/min and 2720 tonne/hr scheduled to be completed in 1979

Note: Normal Maximum Tide Range is 4.9 m

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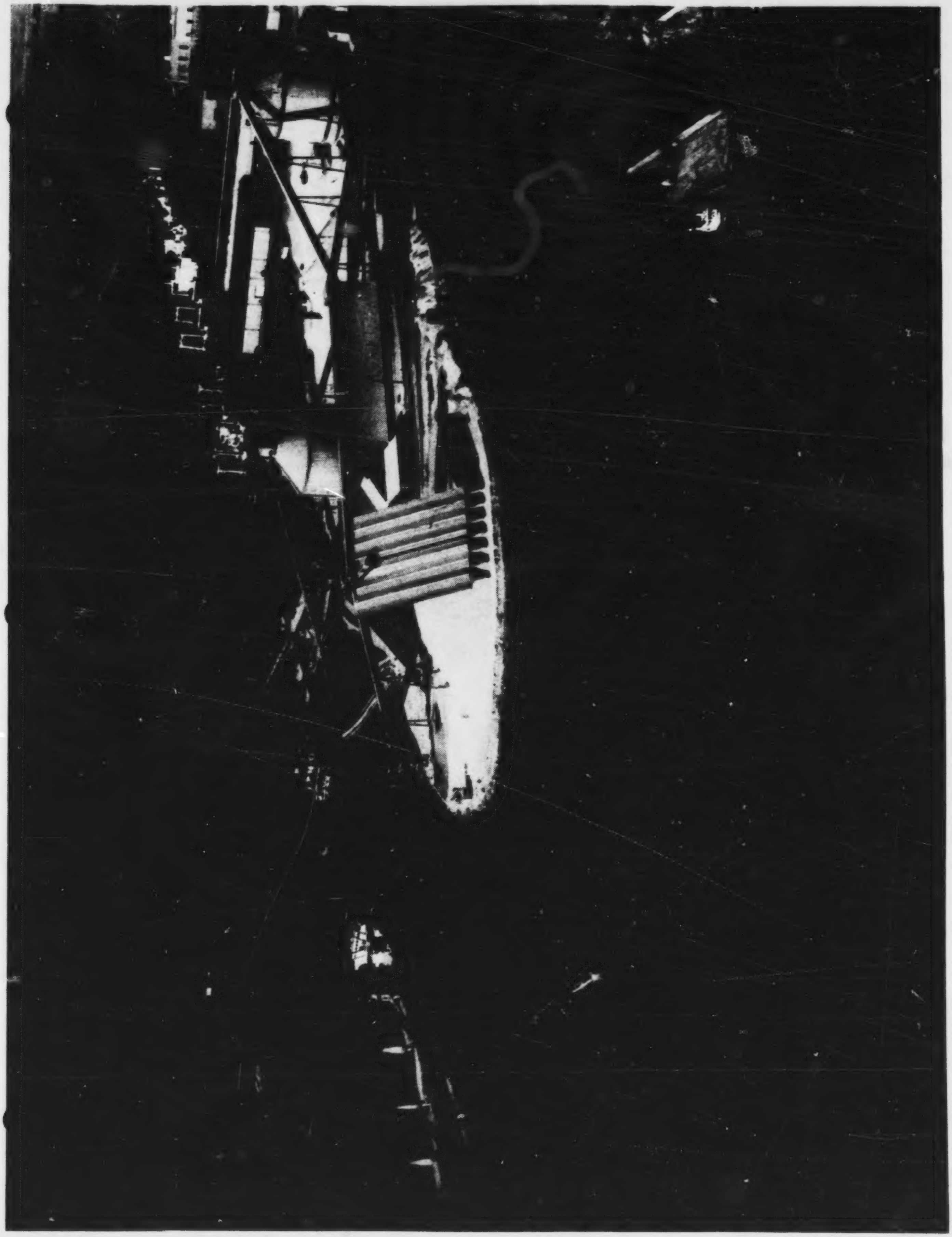


APPENDIX III

REVIEW OF SAMPLING AND CAUSES OF PRODUCT DEGRADATION AT NEPTUNE BULK TERMINAL LTD.

Review of Sampling and Causes of Product Degradation at Neptune Bulk Terminals (Canada) Ltd.

1. Introduction

At the request of Mr. N. Beug, Kalium Canada Ltd., authors: D. Dusterbeck, P. Eng., Process Engineer, Kalium Canada Ltd. and J. Gosselin, Research Chemist, Kalium Canada Ltd. visited Neptune Terminals on October 28, 1987. Objectives of the visit which was made on behalf of the Canpotex Technical Committee were:

1.1 Review Potash product flow paths through the terminal. Examine for points of product degradation and contamination in the handling process. Recommend revisions to the terminal to reduce degradation.

1.2 Review the potash product sampling system and procedures. Recommend alternatives as required. Contacts at Neptune Terminals were Mr. B. S. Sands, Vice President, Operations and Mr. F. Lederer, P. Eng., Director of Facilities Planning.

Sampling and operation of the terminal were discussed with Mr. J. W. Merks, Vice President, Mr. B. B. Singh, President and Mr. R. Annesley, Operations Coordinator, all of PKB Scania, Cargo Surveyors Ltd.

The report contains three sections which correspond to the three sub-systems of the potash handling system. They are as follows:

1. Incoming railcar unloading system. From the railcar hopper through to and including the discharge of No. 3 belt conveyor to the storage building.
2. The storage and reclaim system including the Overhead Feed Belt Conveyor No. 6, warehouse building, reclaim loaders and operating personnel, and the Reclaim Belt Conveyor No. 7.
3. The loadout systems which consist of belt conveyors No. 8 through No. 14, the surge bin and ship loading spouts and trimmers.

2. Considerations in Handling Potash

Potash requires special consideration in handling.

Potash is a friable material with a narrow and critical size range. The generation of fines and dust throughout the product handling system is objectionable. This is true of run of mine floatation products, crystallized product and compacted Granular and Coarse products. With larger products such as Coarse and Granular grades the size is critical because these products are blended with other fertilizer ingredients. If the Fine and Standard products are contaminated with larger particles, complications result to the end user when the product is in solution. Further contamination between grades is objectionable as compacted Granular products will degrade from exposure to anticaking amines applied to Fine and Standard product grades. Products moving through

the terminal are subjected to drops at conveyor transfer points, warehouse, bins, and loading spouts. Graph 1 illustrates the effect of drop height on product breakage degradation. Note that beyond 25-30 feet drop height, breakage degradation does not significantly increase. The level of drops should be limited to less than 10 feet and preferably less than 5 feet. Note also that two drops at >10 feet are worse than a single drop from >50 feet.

Exposure of potash to moisture is objectionable. This is particularly true of compacted Coarse and Granular products. Recent work indicates the level and exposure time to humidity levels above 70% RH cause a reduction in particle competency for these grades. High humidity and/or exposure to moisture will cause portions of a shipment to dramatically shift down in size range as a result of particle breakdown.

3. Considerations in Sampling Potash

Potash sampling may be done manually or by auto samplers and reduction equipment. Sample mass and representation must be agreed upon by the producers, terminal operators, analysis laboratory and customer.

3.1 Auto Sampling

For size analysis samples the recommended suitable dynamic methods for sampling are:

1. Full width linear stream cutter (auto sampler or manual).
2. Hammer sampler on belt conveyor (auto sampler).

Auto sampler design criteria are special for potash. Specifically:

1. Samplers using sliding components and chain drives are not recommended. These mechanisms rapidly become maintenance problems.
2. Internal operating clearances must be generous to avoid binding with built-up material and corrosion products and to allow for easy cleaning.
3. Consumable spare parts such as rubber seals, brushes, etc. are not permitted in the design.
4. Moisture, dust and orphan dirt must not collect in the sample collector.
5. Construction must be robust and simple.

6. Electrical power and control is interfaced with the terminal Allen Bradley PLC Interlock System. Sampling frequency is to be selectable from weigher indication or from programmable timers.
7. The velocity of the sample cutter is to be field adjustable.
8. The sampler must not interfere with conveyor belt maintenance, specifically head pulley and belt replacements.
9. Electrical power and control enclosures to be CEMA 4 dust and water tight.
10. Bearings, actuators and hardware are to be, when possible, duplicates of on site Stores inventory spares.
11. Local lockout is to be provided, safety signs posted and any applicable safety devices or measures required by the terminal operator incorporated in the design.

3.2 Manual Sampling

Manual sampling for full width stream cuts can be made at conveyor head terminal locations. Under continuous operation, vessel and slot sampler units could be used, but would require two employees to physically handle the unit on a 60" belt at 460 ft/min. Aside from being labour intensive, safety aspects and physical access limits manual sampling to selected locations.

4. Incoming Railcar Unloading System

4.1 General

Three railcars at a time can be handled at the railcar unloading pit. Material falls through a space screen into the pit then flood feeds Belt Conveyor No. 1. Belt Conveyor No. 1 reports to Conveyor No. 3 and rises about 75 feet to the top of the storage warehouse tower.

4.2 Sampling

A Denver 68" finger sampler collects a primary increment which reports to a 1.80 Vezin Sample splitter via an integral screw conveyor. The sampler is mounted within the head end hopper of Belt Conveyor No. 3. Purpose of the sampler is to collect samples to prove producer quality assurance requirements have been met.

This sampler is, for the most part, a good example of how not to design a sampler for potash use. Specifically it is plagued by the following design faults.

4.2.1 The design assumes that representation of the entire stream can be made by sampling several thin sections out from the stream then forming a composite primary sample from the thin sections.

4.2.2 The entrance slots for the sampling fingers are not normal to the product stream flow vector. The attitude of the finger slot changes continuously throughout the finger travel.

4.2.3 The sampler moves into the stream, decelerates, stops and accelerates in the reverse direction. During this dwell time for direction reversal, material continuously reports into the sampler slots. This velocity change precludes the possibility of collecting an unbiased sample. Further, the sampler fingers fill to overflowing before completing a full cycle.

4.2.4 The Sample Collection Screw Conveyor integral with the finger shaft conveys the sample out of the headshaft hopper. Use of a screw conveyor on potash samples is not acceptable. Particle attrition within the conveyor will cause particle degradation to shift the entire particle size range finer. This is a critical problem with Coarse and Granular products where the measure of fines must be known with accuracy and precision. Furthermore, there is no provision for cleaning out the conveyor between samples or even between shipments. CEMA Norm 350 specifies 0.5" radial internal clearance for a conveyor of this size. Residual fines in the bottom sector of the screw tube will cause material from sample to report to the next sample or, even worse, between grades and shipments. To further compound problems, dust buildup on the flights and shafting will pollute samples with undesirable fines.

4.2.5 The finger sampler and screw conveyor are driven with oil hydraulic cylinders and motor respectively. A fixed displacement hydraulic system drives the sampler. There is no provision for speed adjustment of the sampler or screw conveyor.

4.2.6 The sampler is difficult to clean.

4.2.7 The authors concur with J. W. Merks' report "Sampling Systems for Potash Terminals at Neptune Terminals", August 30, 1985, Matrix Consultants. In the subject report, Merks maintains the sample mass collected is oversize and the Vezin sampler 1:80 division ratio is too high.

The most logical choice sampler for this position is a linear full stream cut sampler of swinging arm design integral with the hopper housing cover upper part. Proven mechanically reliable designs have been developed.

Sketch SK1 illustrates this design adapted to conveyor No. 3 or No. 8 head terminal end. The sampler consists of two arms; Part "A" pivoting on shaft and bearings "B". The vessel sampler Part "C" is carried in Arms "A". The vessel sampler, rotates in two bearings "D" in the arms. The vessel, axle and adjustable rod "E" are connected to rotate together.

Rod "E" is connected to Rod "G" at yoke and pin connection "F". Rod "G" is connected to a stationary reference frame at "H" which is a shaft on bearings. The linkage system is supplied on both sides of the sampler. All components are inside the housing which picks up bearings "B" and "H". Hydraulic or pneumatic or electric actuators drive the sampler at arms "A".

By arrangement of the links, the sampler transverses the material with the opening normal to the flow vector. When the sampler is past the flow stream, the linkage inverts and dumps the sample. The sampler is parked in the up position and one cycle consists of two grabs; one down and one up. The adjustable length of rods "E" and "G" determine the limit of sampling arc and point to start dumping.

The sampler has the advantage that all parts rotate and are not subject to sliding, binding or rusting up solid. The mechanism is robust in construction. Yokes and hardened pins are NFPA standard items. The vessel is fitted with replaceable and adjustable opening edges. Actuation may use the existing hydraulic cylinders and power unit. The speed of the sampler may be adjusted by changing the radius connection to arms "A" at hydraulic cylinders.

An important feature of the sampler is that it elevates the sample on the return stroke. The collected sample is dumped to a collection hopper integral with the conveyor head end cover.

The sample reports in turn to the existing but modified Vezin divider. Since the divider can be raised on account of the elevated sample dumping point, it is possible to send the Vezin divider sample rejects back into the main material flow in the head terminal cover lower part.

The Vezin divider is to be modified by adding additional collector slots to reduce the splitter ratio to 1:20. This will result in a sample size of 0.28 kg for a primary sample collected with a critically designed linear cutter. It is recommended that the divider be retrofitted with 8 slots; 4 slots at 1.25% area and 4 slots at 0.75% area. Slots can be covered or uncovered as required during field tests to determine the optimum secondary sample size. The Vezin divider drive is satisfactory as is.

4.3 Material Handling

The only objectionable point for product degradation due to impact in the incoming system is the 75 foot approximate warehouse bypass drop. This drop is dead vertical from belt 3 to belt 7. The chute is used for direct hit shipments without flowing thru the warehouse system.

Reduction of product degradation at this point requires elimination of the drop. Quantification of degradation will require sampling and checking -14 mesh fractions on Coarse and Granular products; however, elimination of this drop will be required as a basic terminal improvement.

It is recommended that the chute be revised to slope at 45° from the tower bucket gate to feed belt No. 7. The chute must be free spanning, self supporting design. One feed section and skirtboard must be added to conveyor No. 7 to accommodate the new chute.

An alternate suggestion is to fit the existing vertical chute outlet with a choke feeder gate and level controller which would raise material static head level to conveyor No. 3 head end. Bin wall effects preclude the use of mechanical counterweight choke gates due to the high chute length to area aspect ratio.

5) Storage and Reclaim System

After incoming belt conveyor No. 3 and sampler, material reports to storage feed conveyor 6.

5.1 Sampling

There is no provision for sampling in the warehouse or off of belt conveyors 6 and 7.

5.2 Material Handling

Material degradation and segregation are two problems in the storage system.

Product is dropped approximately 60 feet from No. 6 conveyor tripper to the building floor. Resultant product breakage will produce undesirable fines in the Coarse and Granular grades.

Because product discharges from a single point on the tripper chute, product segregation will occur. This natural phenomenon will cause product fines at the building centerline and at the reclaim feed gates to No. 7 conveyor with coarser product fractions at the building walls. Reclaim loader operators move material to the reclaim gates the easiest and fastest way possible. However, continuous reclaim from one area of the pile for a prolonged period will cause certain portions of a vessel shipment to be off size specification. Sampling operations at outgoing conveyor No. 8 are useful for detecting this type of problem.

Recent work by potash producers has indicated that exposure of compacted products to relative humidities over 70% for a prolonged period will result in breakdown of the compacted particles. Although rain protection is adequate, there is no method for moisture protection. With this facility, it is not possible to close doors during idle periods to limit free air changes. The average annual relative humidity for Vancouver is 81.25%; certainly the RH at harbor side will be higher. All monthly average values are above 70%.

Although the terminal operators exercise care and cleanliness in handling potash, segregation and degradation problems can be partly alleviated by following certain practices. In view of humidity degradation, material must be handled on a first in/first out basis. It is questionable if old warehouse material is moved out first. With direct hit shipments from railcar to vessel flowing through the warehouse building for improved surge capacity.

It is recommended that the terminal operator, potash producers and Canpotex jointly formulate and agree on a practice or specifications for stock rotation on first in/first out basis, for reclaimer operation guidelines and for flowing through or bypassing the warehouse with direct hit shipments.

Product segregation and degradation in the warehouse can be reduced by installation of a segmented launder chute suspended from the warehouse laminated wood beams as shown on sketch SK2. Material flows down each chute which is above the static level of product. The pile is built from one wall towards the opposite wall. At Neptune Terminals the launder would be suspended from the west side of the building so the east doors are always open. An alternative solution is to use a ladder launder as shown in Figures SK3 and SK4. This device distributes to all directions simultaneously about the ladder. Its main feature is that loads are not imposed on the building structure. It is, however, an obstacle for the loader operators and for the free placement of portable partition walls.

6) The Loadout System

6.1 General

The Loadout system starts with conveyor 3 reporting to the surge bin. Material reports to conveyors 9, 10 and 11 and/or 12, 13 and 14 as required to the potash Berth.

6.2 Sampling

The head end of conveyor No. 8 is fitted with a sampler of the same execution as at conveyor No. 3. At this time the sampler is in a state of disrepair and not functional. All discussion and recommendation per section 3 and 4 applies at this point. Sampling the Loadout system at conveyor 8 will be a mandatory requirement for consignee quality assurance tests. Particle size distribution and product abrasion test analysis will be meaningful and representative to the customer if material is sampled at this point only.

6.3 Handling

Product degradation will result from at two points; the surge bin and the vessel loading spout.

A 30 foot (approximate) drop occurs in the surge bin which will cause fines degradation in Granular and Coarse products. The simple solution is for the terminal operator, potash producers and Canpotex to jointly agree that the bin be 80% full at all times. It is desirable to have the bin flowing empty for surge capacity, and if this condition must govern, then the bin should be fitted with a distribution ladder as shown in sketch SK4.

Another significant drop occurs from the vessel loading spout to the hold. The drop height varies according to the adjustable length of the loading spout. There are actually two drops at this location; one from the conveyor head pulley to the spout outlet and another from the spout outlet to the hold. The former can be eliminated by installation of a choke gate and level indicating controller to develop a static head and raised level of material in the feed chute. Neptune Terminals have addressed the use of choke gates prior to this report. Also a great advantage with the choke feeders will be a reduction in product dust when loading.

The use of the belt slinger for handling potash is not recommended. Agreement should be reached to prohibit its use for trimming.

7) Summary Recommendations

7.1 Potash producers, the bulk terminal operator and Canpotex should develop a jointly agreed upon practice for sampling and handling potash through the terminal.

7.2 Detailed engineering and cost estimates be prepared for improved sampling equipment as follows:

  7.2.1 Conveyor No. 3 Head End for incoming products

  7.2.2 Conveyor No. 8 Head End for outgoing products

7.3 A terminal sampling profile program be developed to bias test and new samplers and to quantify product degradation at major drops.

7.4 Detailed engineering and cost estimates be prepared for:

  7.4.1 Relocation of warehouse bypass chute

7.4.2 Installation of warehouse launders

7.4.3 Installation of surge bin ladder.

7.4.5 Table 1 summarizes the above. Suggested priorities are given. An order of magnitude grade cost is also given for each item exclusive of any detail engineering work. Costs are to be used for visibility analysis only.

TABLE 1

SUMMARY OF POSSIBLE IMPROVEMENTS TO NEPTUNE TERMINALS

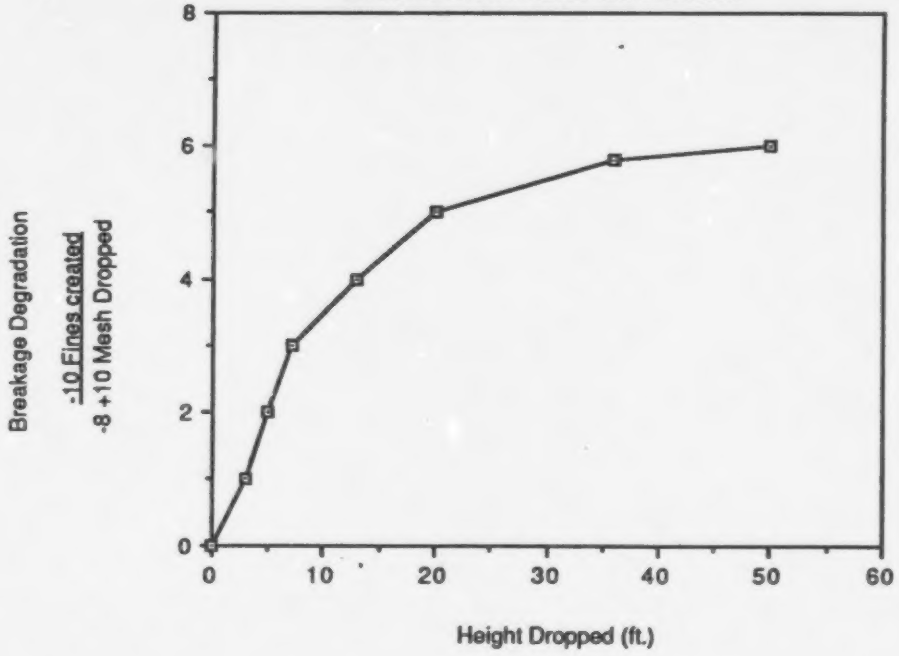
ITEM	FUNCTION	ORDER OF MAGNITUDE DIRECT FIELD COSTS SYSTEMS INSTALLED	SUGGESTED TENTATIVE PRIORITIZATION
1) Modifications to Improve Quality Assurance			
1.1) Install improved sampler at Conveyor No. 3	- Sample incoming material - Ensure quality level conformance of potash producer	15-22 M$	Six: Basis: Quality assurance of incoming material must be known to check supplier quality, humidity degradation, handling degradation
1.2) Install improved sampler at Conveyor No. 8	- Sample outgoing loaded material - Ensure quality level conformance of cargo - Check homogeneous reclaim loader operation	Higher 22-25 M$	Five: Basis: Quality assurance checks to end user must be representative.
2) Modifications to Reduce Product Degradation			
2.1 Install warehouse chute launder or ladder launder system	- Reduce segregation - Reduce product drops	45-55 M$ for 3 qty. launders at third points over warehouse length	Four: Basis: Reducing warehouse drops and segregation. Greater product homogenization during reclaim.
2.2 Install sloped warehouse bypass chute or choke feeder on existing vertical chute	- Reduce product drop in direct shipments when bypassing warehouse	17 - 25 M$	Two: Basis: Product can be circuited thru warehouse
2.3 Install ladder launder in surge bin.	- Reduce product drop and reduce bin segregation while maintaining surge capacity	7 - 9 M$	Three: Basis: Bin can possibly be operated near fill conditions.
2.4 Install loading spout choke feeders	- Reduce product drop and reduce dust while loading	Completed by Canpotex	One: Basis: Environmental concerns regarding loading dust and reducing product breakage

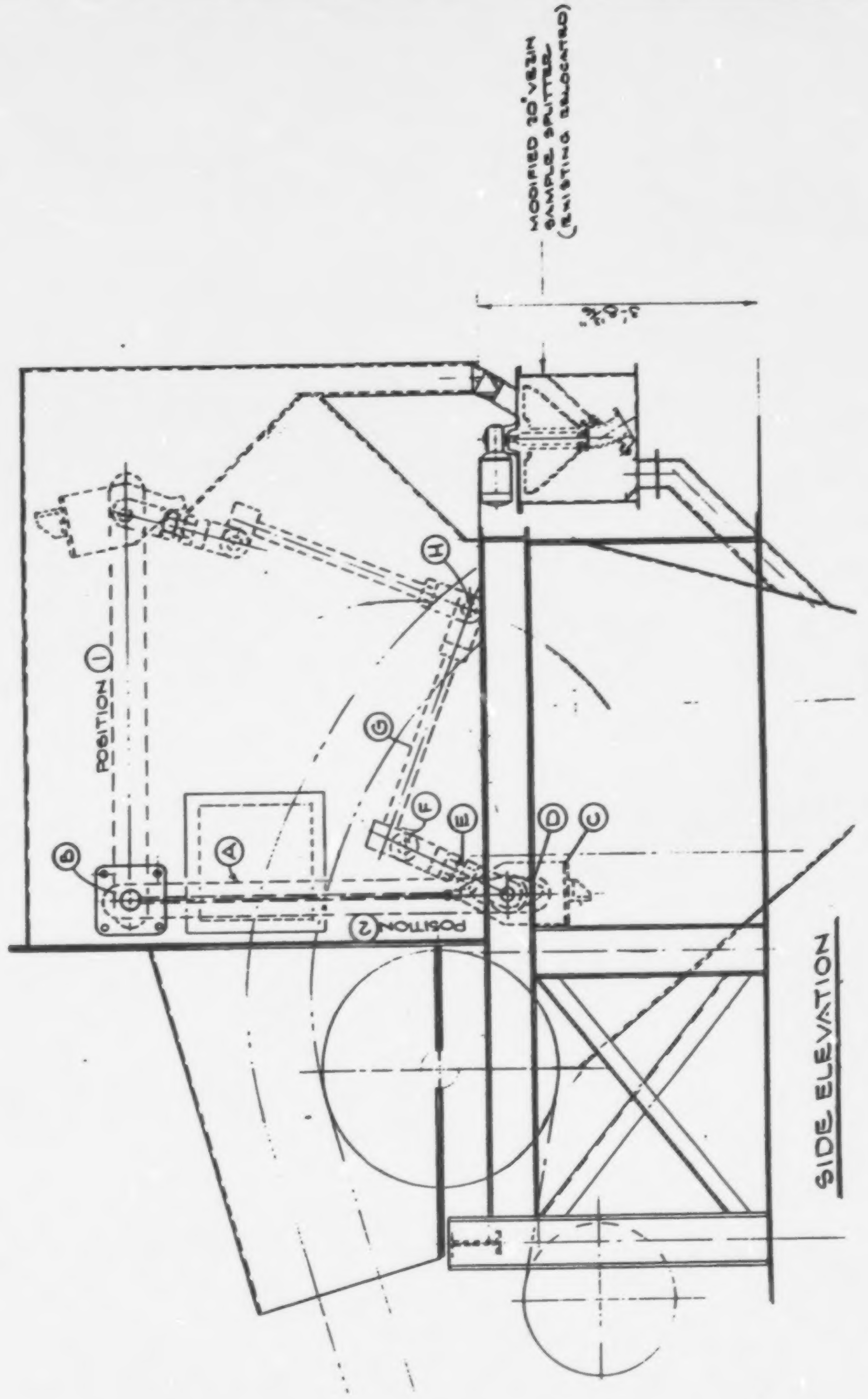
RJAG/11/28/88 TAB

Graph 1

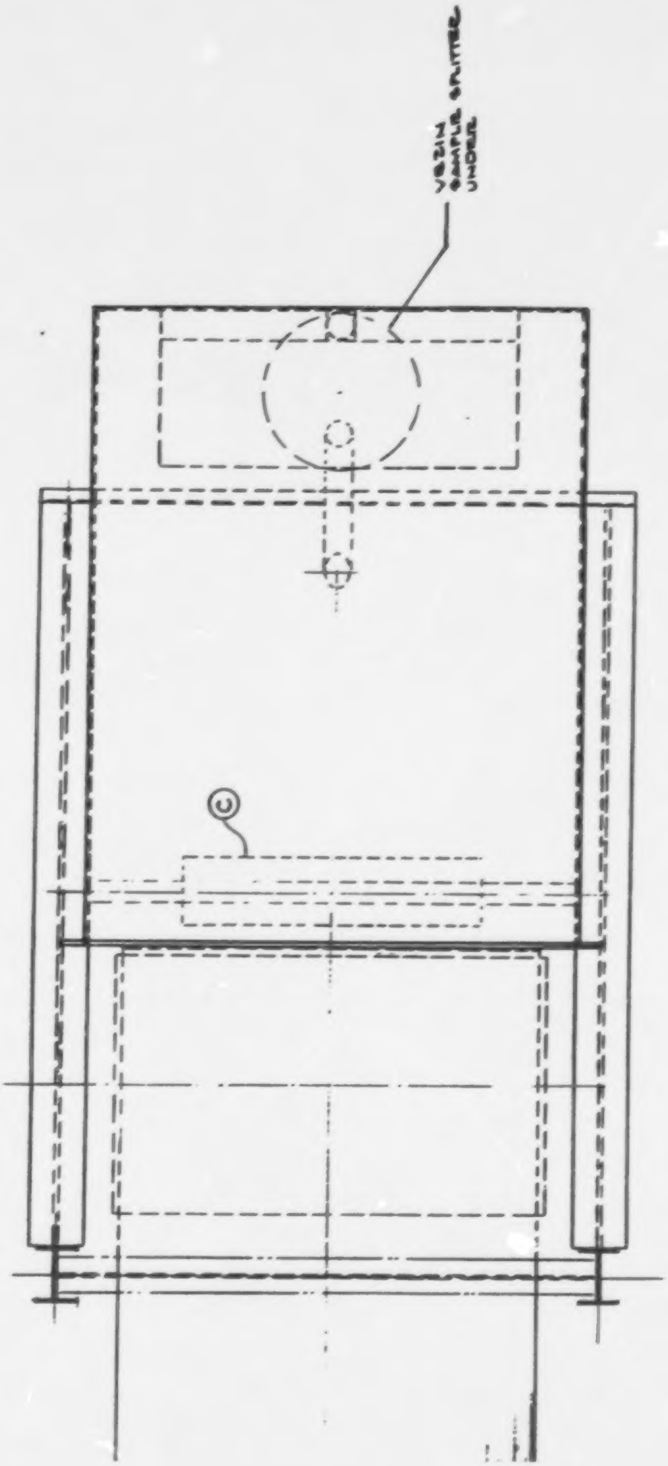
EXAMPLE OF PRODUCT BREAKAGE

Curve may vary depending upon Crystallizer, Mill run or Compacted Processing Method, the Producer, previous storage conditions and en-masse flow conditions




MODIFIED 20" VEZIN
SAMPLE SPLITTER
(EXISTING RELOCATED)
3'-0¾"
H
POSITION ①
G
F
E
D
C
B
A
POSITION ②
SIDE ELEVATION

JOB No.			**KALIUM CHEMICALS** A DIVISION OF PPG CANADA INC.
DRAWN	BC	18-NOV-87	
CHECKED			TITLE - NEPTUNE TERMINALS
APPRV'D			SWING ARM SAMPLER · SIDE ELEVATION
SCALE			DRG. No.- SK1-A REV.


VEZIN SAMPLE SPLITTER UNDER
C

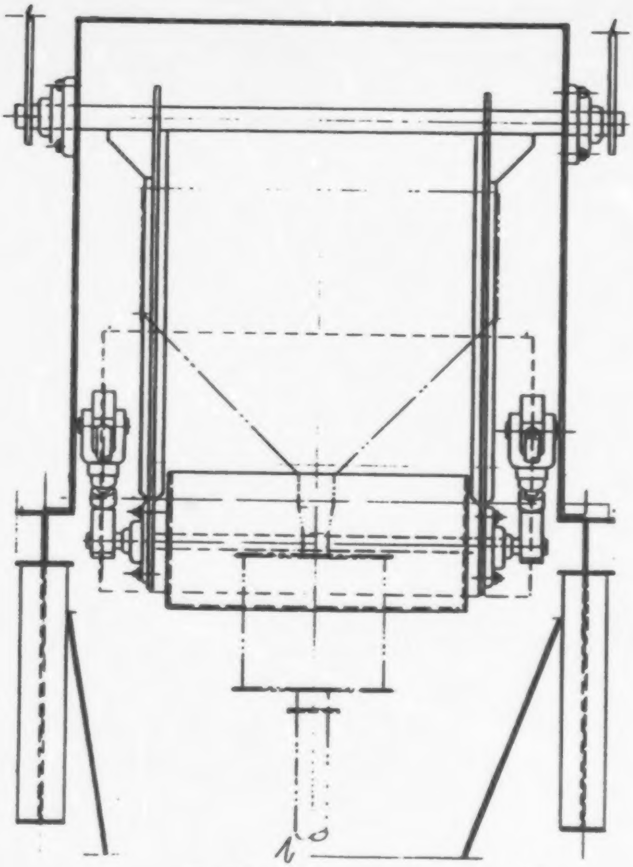
PLAN VIEW

JOB No.		
DRAWN	BC	18-NOV-87
CHECKED		
APPRV'D		
SCALE		

KALIUM CHEMICALS
A DIVISION OF PPG CANADA INC.

TITLE- NEPTUNE TERMINALS
SWING ARM SAMPLER - PLAN VIEW

DRG. No.- SK1-B REV. △

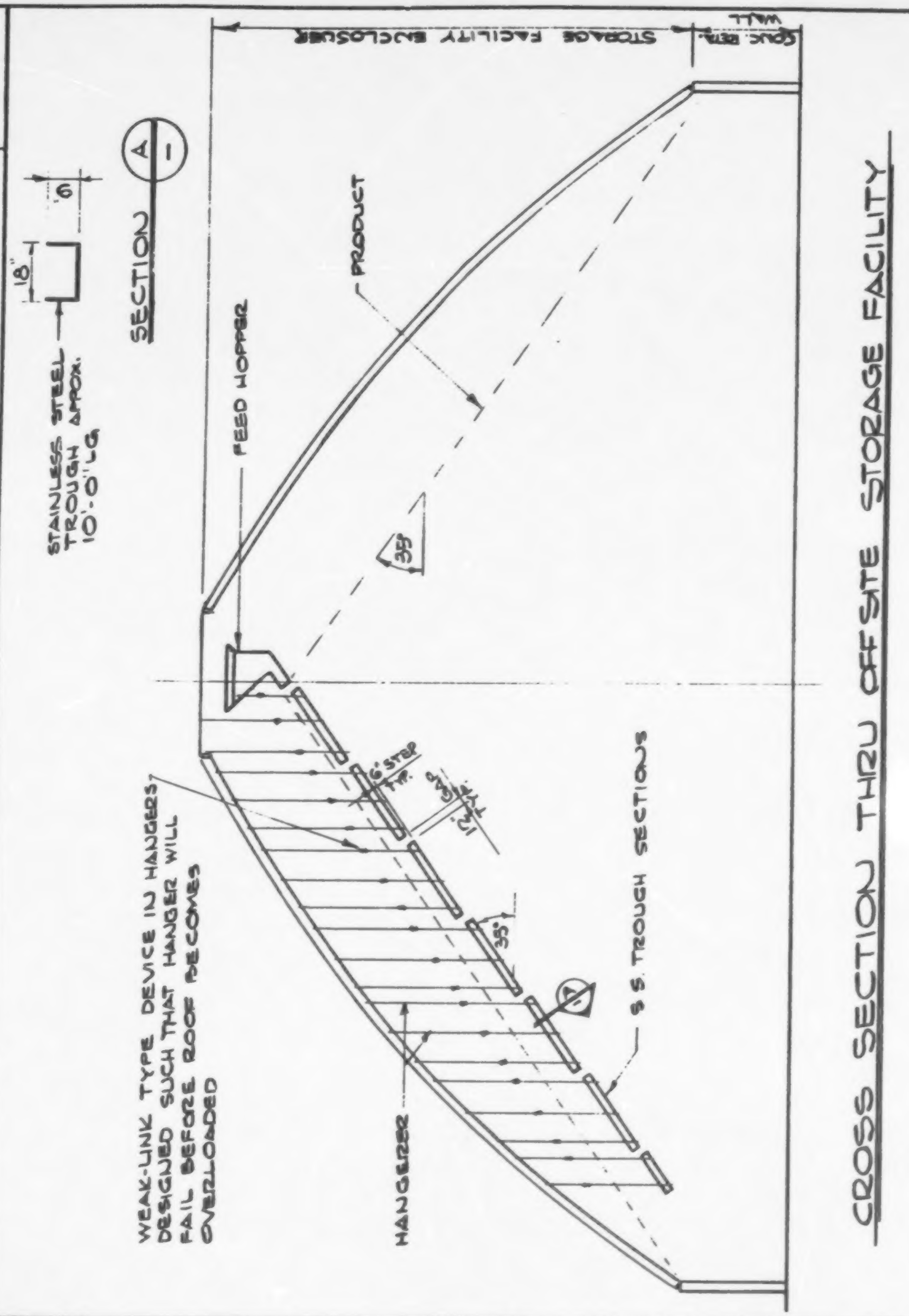


END VIEW -(CUTAWAY)

JOB No.		
DRAWN	BC	18·NOV·87
CHECKED		
APPRV'D		
SCALE		

KALIUM CHEMICALS
A DIVISION OF PPG CANADA INC.

TITLE - NEPTUNE TERMINALS
SWING ARM SAMPLER - END VIEW
DRG. No.- SK1-C
REV.


CONC. RET. WALL
STORAGE FACILITY ENCLOSURE
PRODUCT
FEED HOPPER
35°
6" STEP TYP.
GAP 1/2" TYP.
S.S. TROUGH SECTIONS
35°
A
1
HANGERS
WEAK-LINK TYPE DEVICE IN HANGERS DESIGNED SUCH THAT HANGER WILL FAIL BEFORE ROOF BECOMES OVERLOADED
STAINLESS STEEL TROUGH APPROX. 10'-0" LG.
18"
9"
SECTION A
1

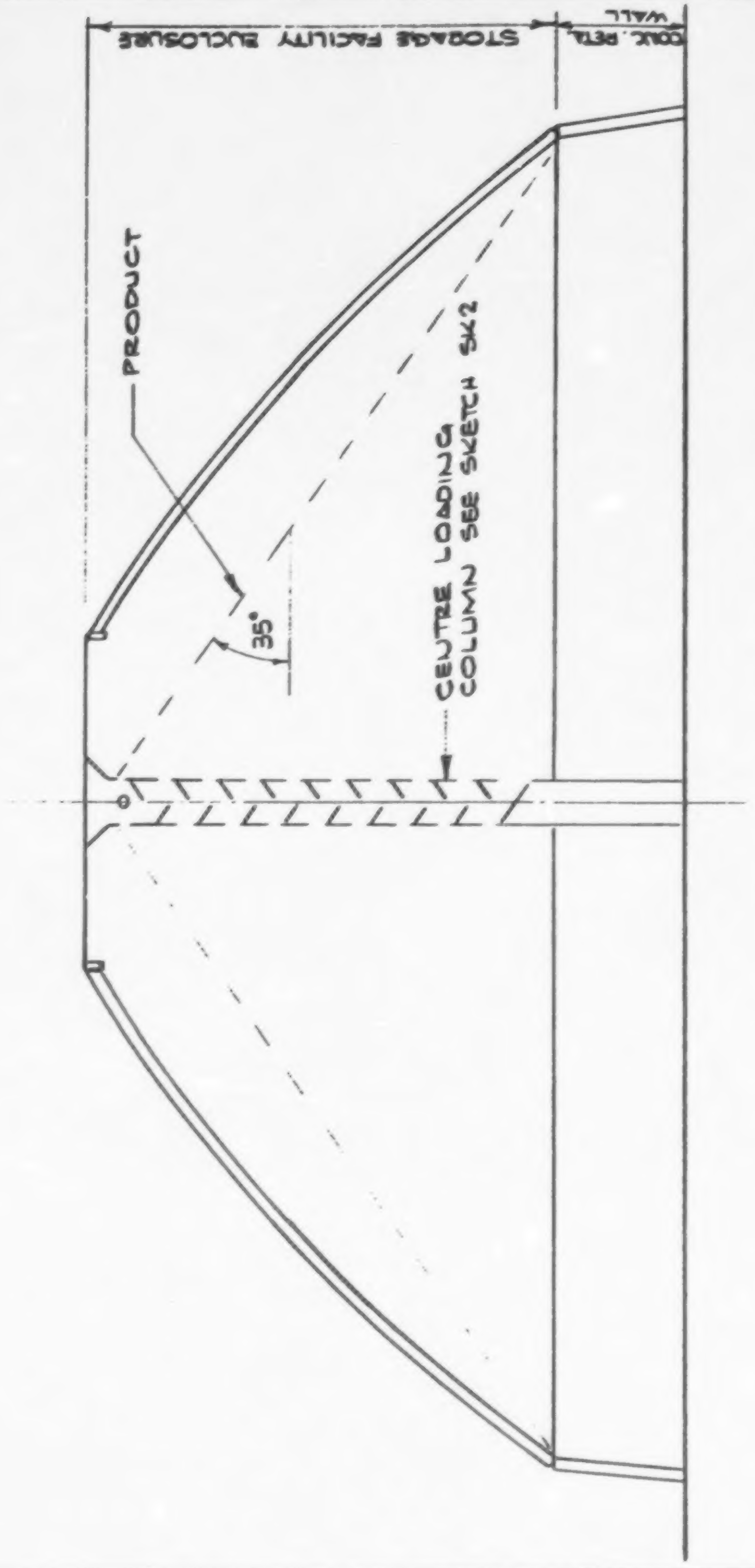
CROSS SECTION THRU OFFSITE STORAGE FACILITY

JOB No.		
DRAWN	K. FUESSEL	8-APR-88
CHECKED		
APPRV'D		
SCALE		

KALIUM CHEMICALS
A DIVISION OF PPG CANADA INC.

TITLE - NEPTUNE TERMINALS
SEGMENTED LAUNDER CONCEPT SKETCH

DRG. No.- SK2
REV.


STORAGE FACILITY ENCLOSURE
CONC. RETL. WALL
PRODUCT
35°
CENTRE LOADING COLUMN SEE SKETCH SK2

CROSS SECTION THRU OFFSITE STORAGE FACILITY

JOB No.			**KALIUM CHEMICALS** A DIVISION OF PPG CANADA INC.
DRAWN	K. FUESSEL	8-APR-88	
CHECKED			TITLE- NEPTUNE TERMINALS
APPRV'D			LADDER LAUNDER CONCEPT SKETCH
SCALE			DRG. No.- SK3 REV.

NEPTUNE BULK TERMINALS (CANADA) LTD.

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North Vancouver, B.C.
Telephone (604) 985-7461
Telex 04-352531

NEPTUNE BULK TERMINALS (CANADA) LTD.

DRY BULK SYSTEM WASHDOWN PROCEDURE

The variety of products being handled on the dry bulk system requires the system to be cleaned between products. The following clean-up guide will prevent any contamination between products:

"CARS TO STORAGE"

1. Dumper

   - Top rail area - under rail flange.
   - Side floor and ditches - behind lids.
   - Go down ladder into dumper pit - ends and sides must be washed.
   - #1 must be run on fast speed to get water off belt.
   - Wash out headbox of #1.

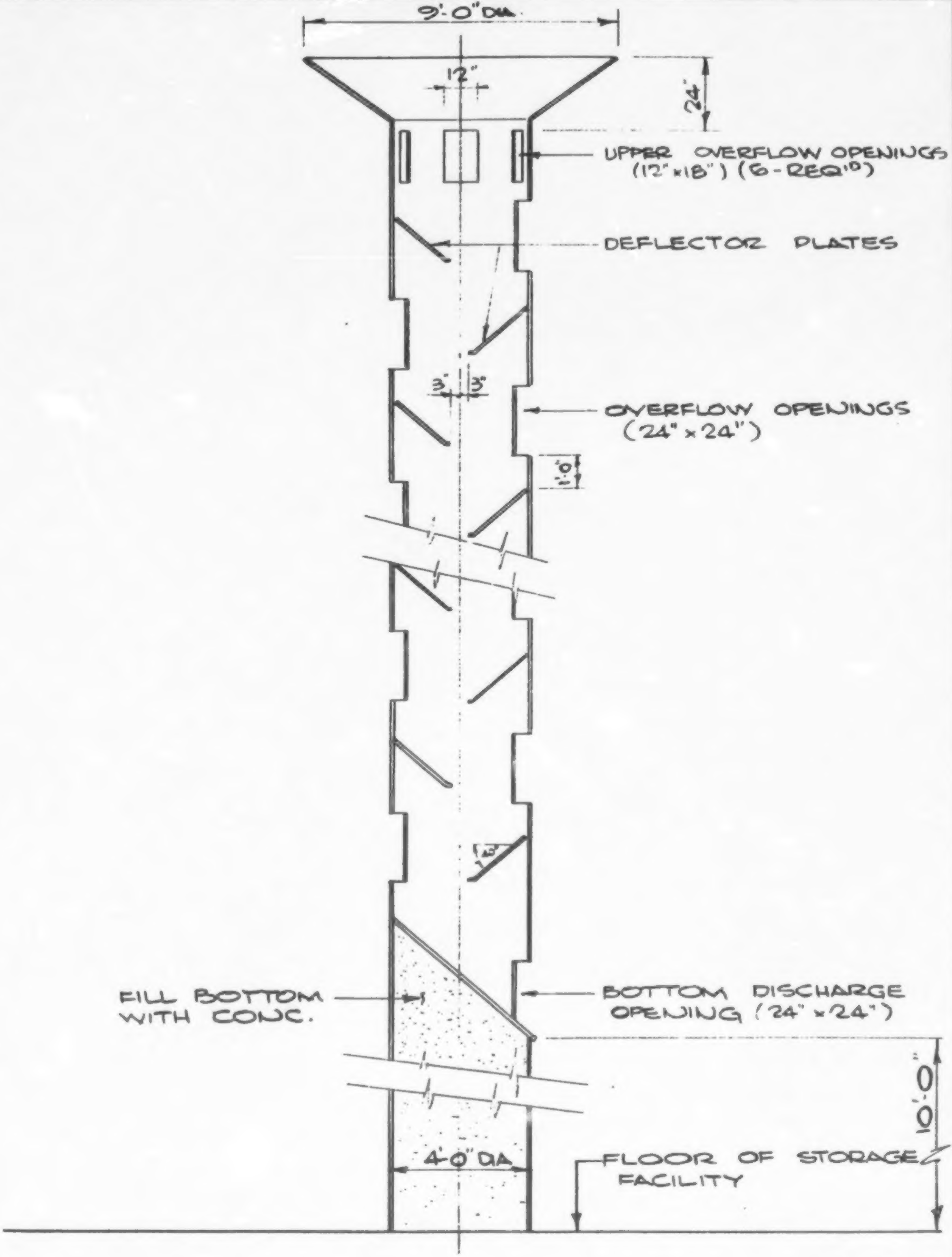
2. #3 Conveyor

   - Tail plate must be open - wash off tail end.
   - Belt must be "laundered" if there is an accumulation of "pellet" dust stuck to belt.
   - Head box and around sampler sealing pad must be washed.

3. #6 Conveyor

   - Tail plate must be open - wash off tail end.
   - Spot tripper over an empty bay or a pellet bay if cleaning up after pellets.
   - Check inside tripper headbox chute for any material "hanging up" and sweep if necessary.
   - All belts must be run off before moving tripper to proper bay.

System must be thoroughly inspected by supervisor in charge before any cargo is handled.


9'-0" DIA
12"
24"
UPPER OVERFLOW OPENINGS
(12"×18") (6-REQ'D)
DEFLECTOR PLATES
3" 3"
OVERFLOW OPENINGS
(24" × 24")
1'-0"
45°
FILL BOTTOM
WITH CONC.
BOTTOM DISCHARGE
OPENING (24" × 24")
10'-0"
4'-0" DIA
FLOOR OF STORAGE
FACILITY

JOB No.			**KALIUM CHEMICALS** A DIVISION OF PPG CANADA INC.
DRAWN	K. FUESSEL	8-APR-88	
CHECKED			TITLE- NEPTUNE TERMINALS
APPRV'D			LADDER LAUNDER DETAIL SKETCH
SCALE			DRG. No.- SK4 REV.

APPENDIX IV

NEPTUNE BULK TERMINALS LTD
DRY BULK SYSTEM WASHDOWN PROCEDURE

NEPTUNE BULK TERMINALS (CANADA) LTD.
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NEPTUNE BULK TERMINALS (CANADA) LTD.

DRY BULK WASH-DOWN PROCEDURE

The variety of products handled on the dry bulk system requires the system to be cleaned between products. The following clean-up guide will prevent any contamination between products.

"STORAGE TO VESSEL" and "CARS TO VESSEL"

(See also "Cars to Storage")

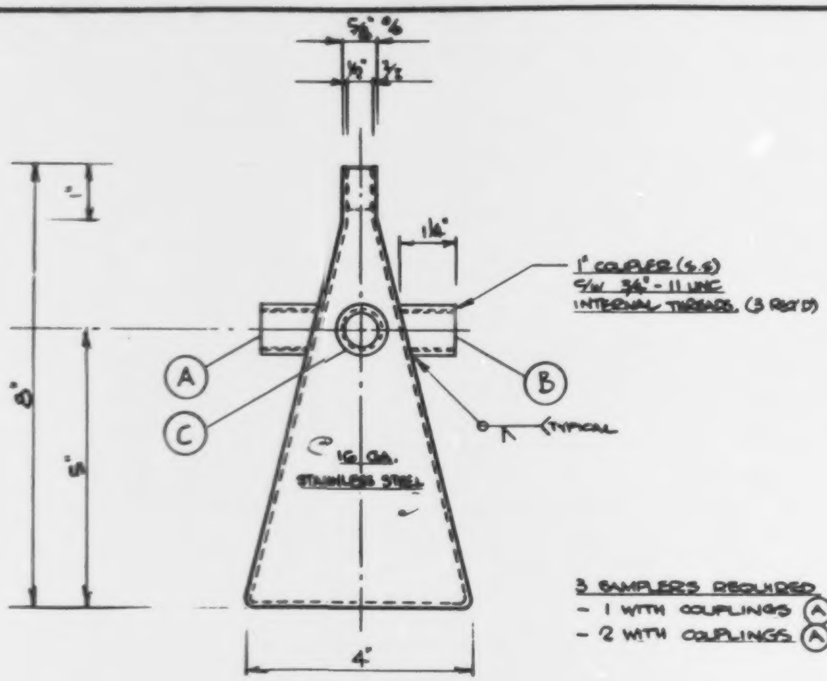
- Surge tank gates, all tail plates, all inspection doors to be opened and scrapers to be released.

The following to be washed:

- #3 - #7 bypass and small surge tank.
- Entrance and bypass transfer in #7 tunnel.
- "Run-off" #7 belt.
- #7 - #8 transfer behind scraper.
- Head box of #8 inside surge tank including roof.
- Pantlegs, using internal sprays.
- Tail end of #9 and #12 including behind baffles.
- Head box of #9 and #12 including behind scrapers.
- Bottom of #9-#10 and #12 - #13 transfer. Look in inspection holes.
- Tail end of #10 and #13 including behind baffles.
- Roof over #10 and #13.
- Head box of #10 and #13 including behind scrapers.
- Head box of #11 and #13 including behind scrapers.
- Wash down telescopic spout while standing at the head end of boom.
- Wash off both trimmers, pay attention to motor bases.
- Run off all belts with all spray bars on and let run for 15 minutes.
- System is now ready for inspection.
- On completion of inspection, close up all tail plates, inspection doors, and tighten scrapers.
- Run all belts for about 15 minutes to dry off before handling cargo.

APPENDIX V

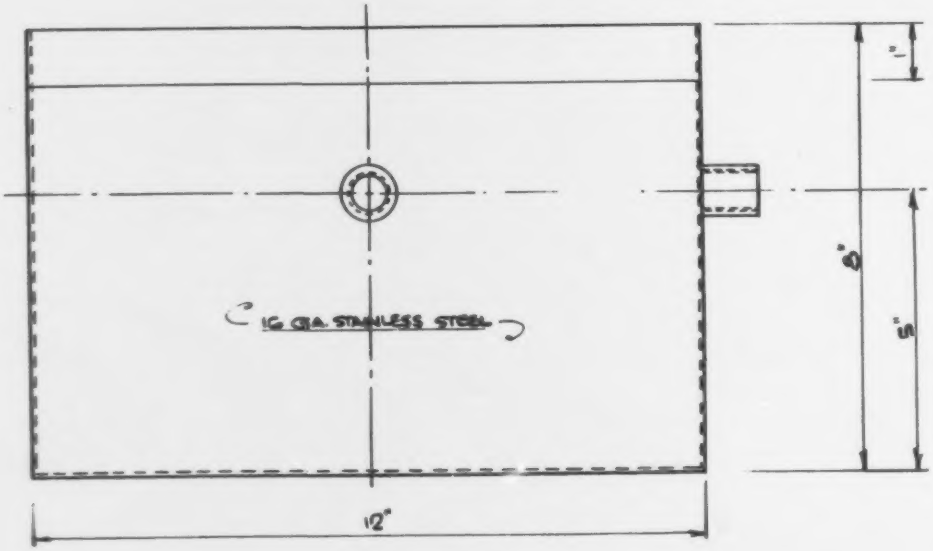
DRAWINGS OF SAMPLING EQUIPMENT


5/8" 1/16
1/2" 3/32
1"
8"
5"
1 1/4"
4"
A
B
C
16 GA. STAINLESS STEEL
TYPICAL

1" COUPLER (S.S) C/W 3/4" - 11 UNC INTERNAL THREADS. (3 REQ'D)

3 SAMPLERS REQUIRED
- 1 WITH COUPLINGS (A), (B) & (C).
- 2 WITH COUPLINGS (A), (B).

SIDE

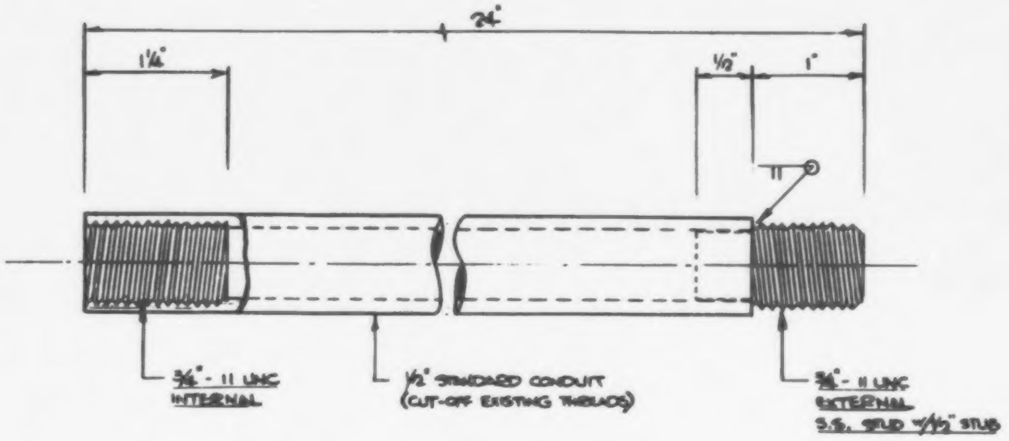

1"
8"
5"
12"
16 GA. STAINLESS STEEL

FRONT

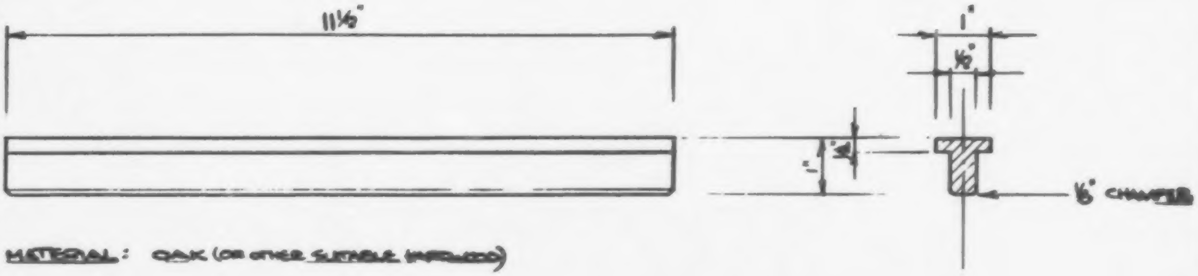
- WELD ALL CORNERS - GRIND & POLISH SMOOTH
- REMOVE ALL POINTS.

SAMPLER

JOB No.			KALIUM CHEMICALS
DRAWN	FS		
CHECKED			TITLE- PORTABLE SAMPLER
APPRV'D			SIDE & FRONT VIEW
SCALE			DRG. No.- REV.


24"
1¼"
½"
1"
¾" - 11 UNC
INTERNAL
½" STANDARD CONDUIT
(CUT-OFF EXISTING THREADS)
¾" - 11 UNC
EXTERNAL
S.S. STUD W/½" STUB

HANDLE (EXPANDABLE) 6 (REQ'D)


11½"
1"
½"
1"
¾"
⅛" CHAMFER

MATERIAL: OAK (OR OTHER SUITABLE HARDWOOD)

HARDWOOD PROTECTOR STRIP FOR SAMPLER OPENING

JOB No.		
DRAWN	PS	
CHECKED		
APPRV'D		
SCALE		

KALIUM CHEMICALS

TITLE- PORTABLE SAMPLER
HANDLE & PROTECTOR

DRG. No.- REV.

APPENDIX VI

PROCEDURE FOR SAMPLING THE UNLOADING OF INDIVIDUAL RAILCARS

Procedure for Sampling the Unloading of Individual Railcars

This procedure is not to be used for the sampling of all railcars within a unit train but to spot check a few railcars.

Sampling

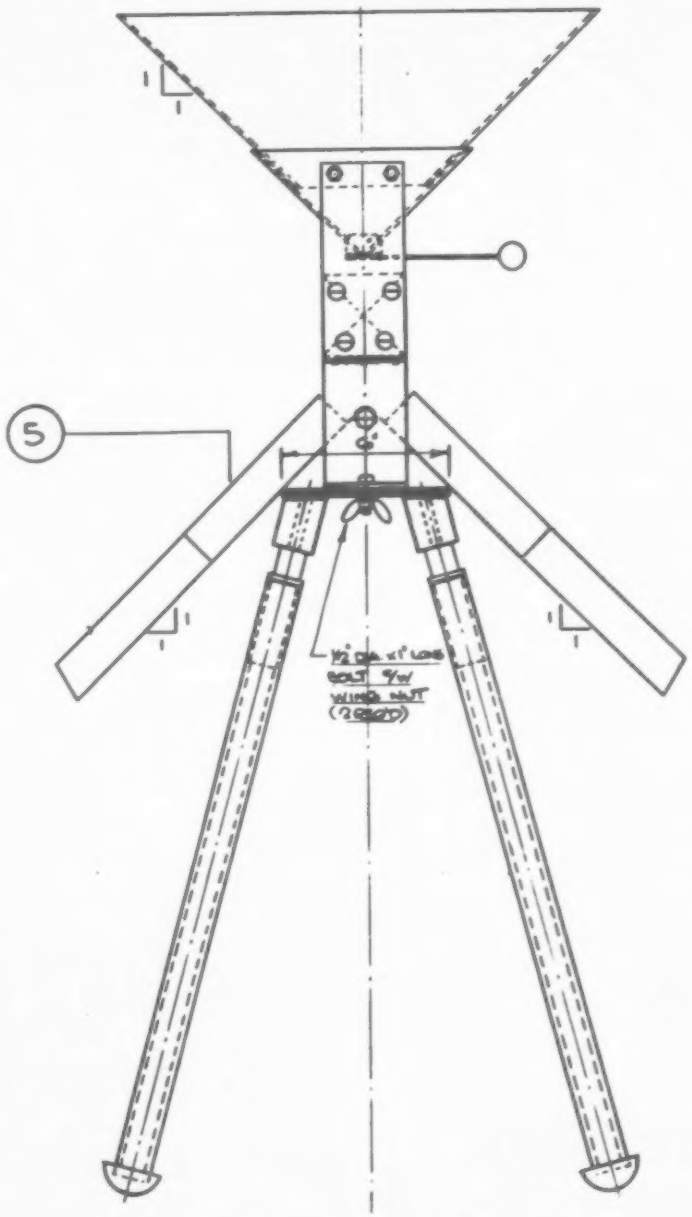
1. Open one hopper car gate 6 to 8 inches.
2. Ensure steady and constant flow is coming out of the railcar.
3. With the manual full stream sampler cut the stream with a steady and regular stroke. One person is pushing while the other is pulling. Ensure that the sampler does not overfill each time the stream is cut and that the speed of the sampler does not exceed 0.6 ml.
4. Pour sample into pail. Pails can be on either side or one side of truck.
5. Keep cutting stream at constant intervals of 1 to 2 minutes until hopper is empty.
6. Open next gate and start at step 1.

Sample Reduction

1. Pour sample into hopper of modified gated riffle.
2. Open gate and let product flow through.
3. Reject one portion.
4. Save other portion and return to step 2 until desired weight (5 to 10 lbs) is achieved.
5. Carefully store and transport sample in a sealed plastic bag, or container.

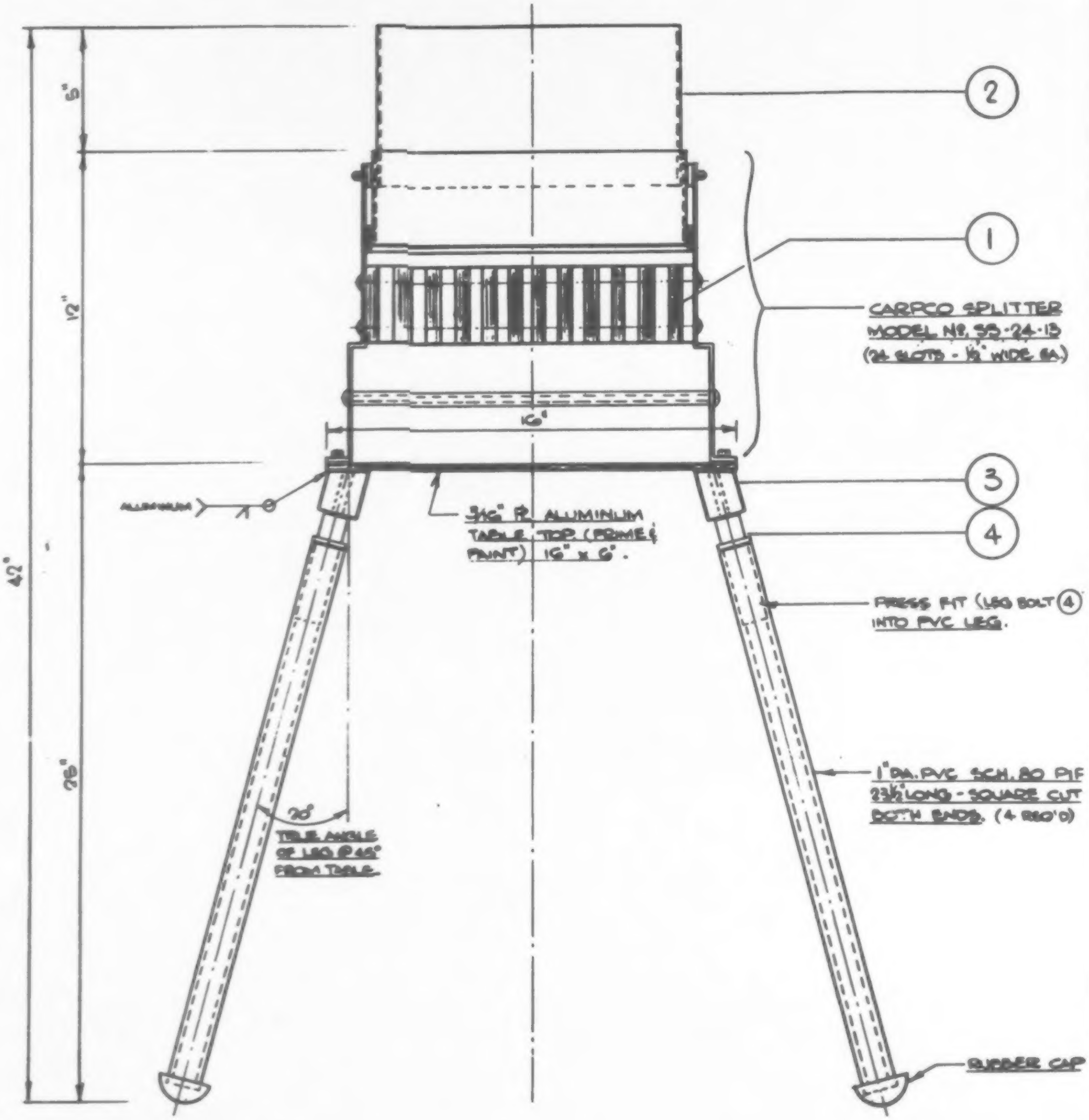
APPENDIX VII

DRAWINGS OF PORTABLE SAMPLE SPLITTER


5
1/2" DIA x1" LONG BOLT c/w WING NUT (2 REQD)

SIDE ELEVATION

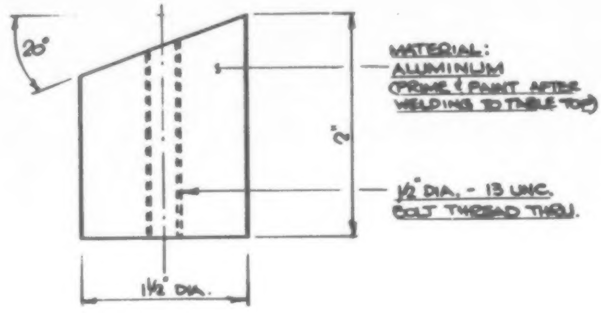
JOB No.			**KALIUM CHEMICALS**
DRAWN	PS		TITLE - PORTABLE SAMPLE SPLITTER
CHECKED			SIDE ELEVATION
APPRV'D			
SCALE			DRG. No.- REV.



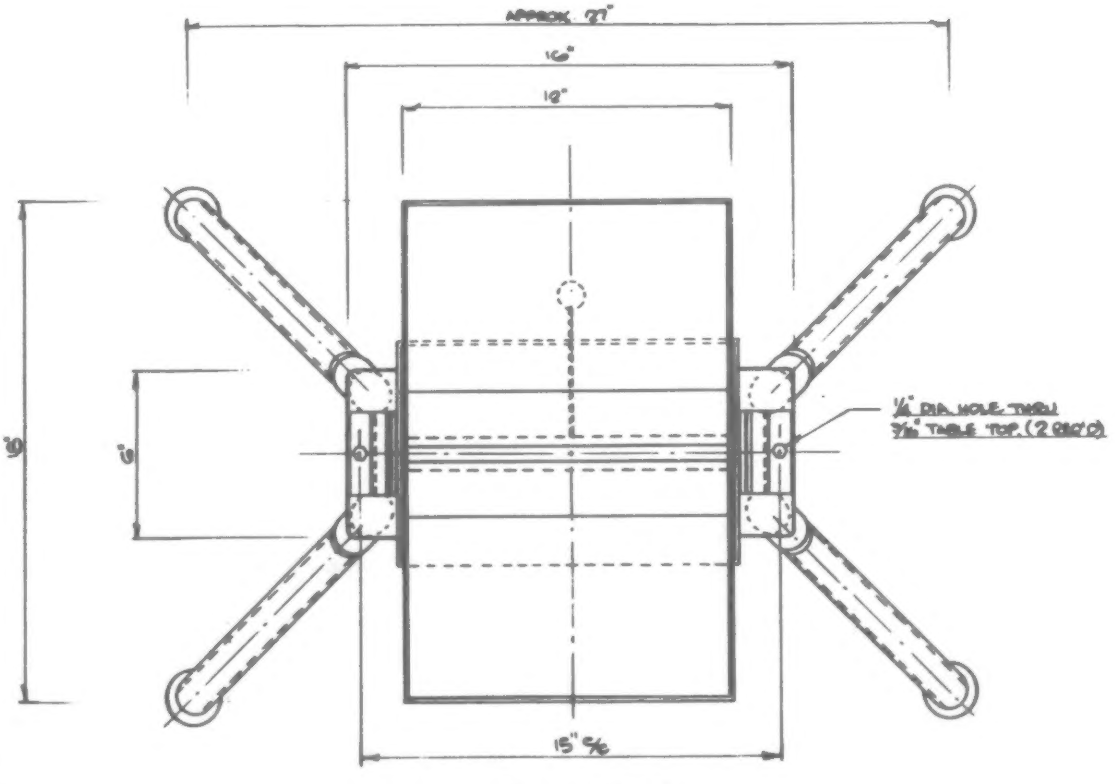
5"
12"
42"
25"
16"
②
①
③
④
CARPCO SPLITTER
MODEL Nº. SS-24-13
(24 SLOTS - ½" WIDE EA.)
ALUMINUM
3/16" P. ALUMINUM
TABLE TOP (PRIME &
PAINT) 16" x 6".
PRESS FIT (LEG BOLT ④
INTO PVC LEG.
20°
TRUE ANGLE
OF LEG @ 45°
FROM TABLE.
1" DIA. PVC SCH. 80 P.I.F
23½" LONG - SQUARE CUT
BOTH ENDS. (4 REQ'D)
RUBBER CAP

FRONT ELEVATION

JOB No.		KALIUM CHEMICALS
DRAWN	PS	
CHECKED		TITLE- PORTABLE SAMPLE SPLITTER
APPRV'D		FRONT ELEVATION
SCALE		DRG. No.- REV.

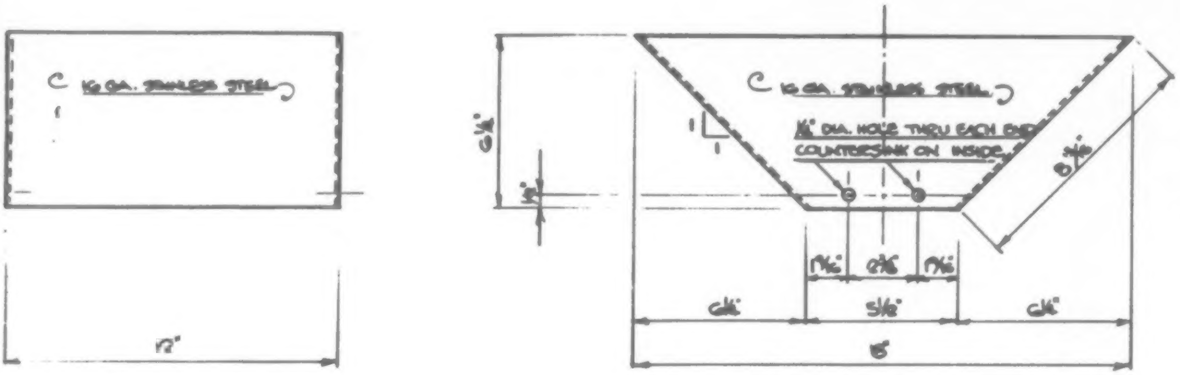

20°
MATERIAL:
ALUMINIUM
(PRIME & PAINT AFTER
WELDING TO TABLE TOP)
2"
1/2" DIA. - 13 UNC.
BOLT THREAD THRU.
1 1/2" DIA.

LEG COUPLER DETAIL ③


APPROX. 27"
16"
18"
30"
6"
1/4" DIA. HOLE THRU
7/16" TABLE TOP. (2 REQ'D)
15" 5/16

TOP VIEW

JOB No.			KALIUM CHEMICALS
DRAWN	PS		
CHECKED			TITLE - PORTABLE SAMPLE SPLITTER
APPRV'D			TOP VIEW & DETAIL
SCALE			DRG. No.- REV.

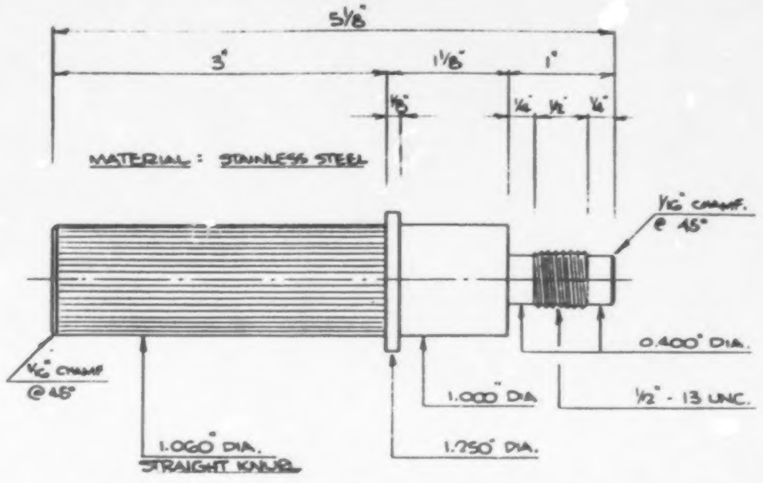

16 GA. STAINLESS STEEL
1/4" DIA. HOLE THRU EACH END
COUNTERSINK ON INSIDE.
12"
15"

FRONT

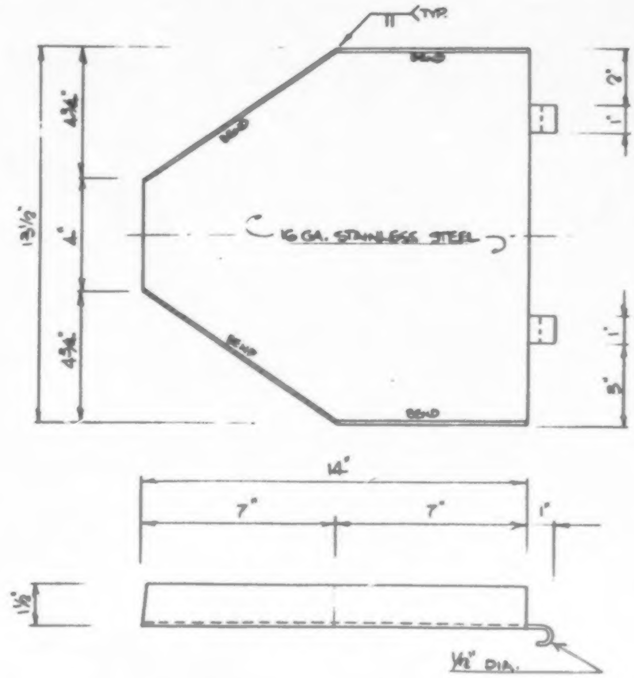
SIDE

TOP HOPPER DETAIL ②

JOB No.			**KALIUM CHEMICALS**
DRAWN	PS		
CHECKED			TITLE - PORTABLE SAMPLE SPLITTER
APPRV'D			HOPPER DETAIL
SCALE			DRG. No.- REV.


5⅛"
3"
1⅛"
1"
⅛"
¼"
½"
¼"
MATERIAL: STAINLESS STEEL
1/16" CHAMF. @ 45°
0.400" DIA.
1/16" CHAMF @45°
1.000" DIA
½" - 13 UNC.
1.060" DIA. STRAIGHT KNURL
1.250" DIA.

LEG BOLT DETAIL ④


TYP.
BEND
BEND
BEND
BEND
13½"
4¾"
4"
4¾"
2"
1"
1"
3"
16 GA. STAINLESS STEEL
14"
7"
7"
1"
1½"
½" DIA.

- GRIND & POLISH ALL WELDS SMOOTH.
- REMOVE ALL SHARP EDGES & POINTS

PAN DETAIL ⑤

JOB No.			**KALIUM CHEMICALS**
DRAWN	PS		
CHECKED			TITLE - PORTABLE SAMPLE SPLITTER
APPRV'D			PAN & BOLT DETAIL
SCALE			DRG. No.- REV.

APPENDIX VIII

CARPCO PRECISION SAMPLE SPLITTERS

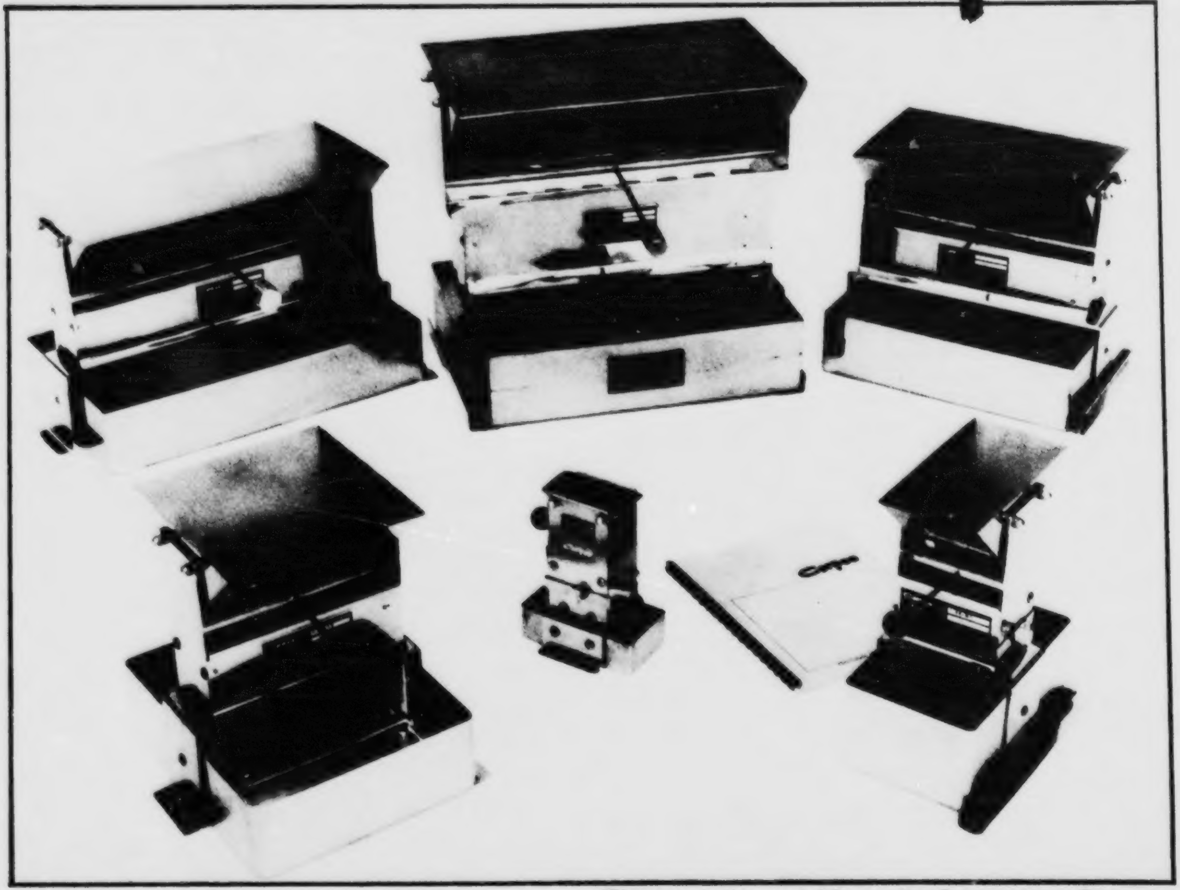
NEW MICROSPLITTERS

Precision Sample Splitters

Rifflers for:

- Ores
- Fertilizers
- Chemicals
- Pharmaceuticals
- Seeds
- Granular Mixtures

Carpco®



BULLETIN 8490 © Carpco, Inc

Electrostatic and Magnetic Separation Systems

Carpco®

Introduction

Carpco sample splitters are classified as precision 'rifflers' because of their ability to accurately divide granular materials for sampling purposes. Top ranked by material testing organizations, Carpco sample splitters meet Section 5 of ASTM Specification C702, 'Standard Methods for Reducing Field Samples of Aggregate to Testing Size,' and other sampling specifications. Precision splitting or riffling is accomplished simply by loading the hopper with material to be divided, leveling sample and lifting the palm-operated gate release. Alternating fixed-width chutes deliver half the sample to each pan. The hopper-gate mechanism assures even sample distribution and eliminates particle segregation.

In **standard** models, hopper, gate, splitter blades and side walls are of polished stainless steel construction. Chute bottoms and product pans are of a wear-resistant aluminum alloy; the exception to this is Model SS-24-13 which is supplied with stainless steel pans.

All-stainless construction models are also available that have special appeal to fertilizer, powdered metals, nuclear and other specialty industries.

Construction of all models is of a heavier gauge and is more exact than splitters from other sources.

Applications

	Recommended Models
• Grain Counting by Geologists and Mineralogists	SS16-3, SS24-1.5
• Fertilizer Sampling	SS24-13, SS24-13X
• Heavy Metals or Nuclear Materials Analyses	SS16-3X, SS16-6X
• Powdered Metal Analysis	SS16-3X, SS16-6X, SS32-6X
• Sampling of Beach Sands and Other Alluvials	SS32-6, SS64-6
• Iron Ore Assay Preparation	SS16-13
• Chopped Wire and Plastic Sampling	SS16-13, SS32-13, SS16-25
• Seeds and Foodstuffs Sampling	SS16-13, SS32-13, SS16-25

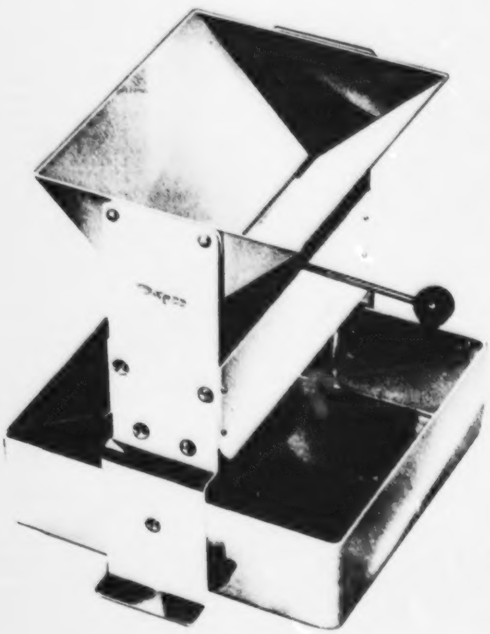
Features

Feed Hopper is constructed of polished heavy gauge stainless steel. Refer to "Specifications" table on back page of hopper capacities.

Palm-operated gate mechanism is designed not only to minimize particle segregation but also to eliminate the need for a third product pan.

The precision **hopper slot** is designed to correspond to a specific splitter slot width.

Precision **splitter slots** are of aluminum alloy coupled with stainless steel splitter blades. Splitters with slots of all-stainless steel are available on "all-stainless construction" models. Recommended slot sizes and corresponding models are shown in Table 1.



Sample pans for standard models are of drawn aluminum alloy except for Model SS-24-13 which has stainless steel pans. This model is supplied with stainless pans because of its wide use in corrosive fertilizer sampling. (See "Specifications" on the back cover.) Pans for all-stainless models are of stainless steel construction.

Electrostatic and Magnetic Separation Systems

Microsplitters

Carpco's popular **microsplitter** series is now being offered in two versions. Carpco Microsplitter Model SS-16-3 has 16 slots, 3 mm (1/8 inch) wide; and super-precision Microsplitter Model SS-24-1.5 has 24 slots, 1.5 mm (1/16 inch) wide, for improved statistical division. The precision microsplitter is used by professionals in many areas such as grain-counting or ore analysis where minute quantities or exacting quantitative results are involved. The microsplitter is offered in both standard and all-stainless constructions.

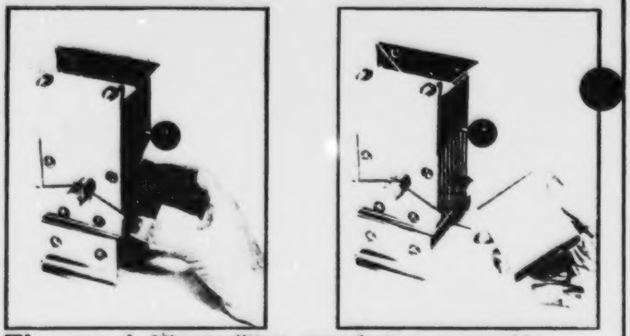


Figure 1 Microsplitters now feature removable dust shields for ease of cleaning.

Sampling Procedure

1. Determine minimum final sample size for end use based on particle size, composition, liberation and specific gravity.
2. A representative portion of the parent lot of material to be tested must be obtained by appropriate sampling procedure.
3. Select an appropriate sample splitter from Table 1 based on the quantity of material to be handled and particle size. Some materials such as coarse run-of-mine ores will necessitate large minimum sample quantities which will have to be reduced in particle size by crushing and/or grinding, and then riffled to obtain the desired representative end sample.
4. Figure 2 illustrates a typical sampling procedure for sampling splitting or riffling. Note that opposite cuts are taken to avoid possible splitting bias.
5. Clean the sample splitter with an air hose or by brushing between different samples to avoid contamination.

Figure 2

Sample Splitting Diagram
Total Sample = 100

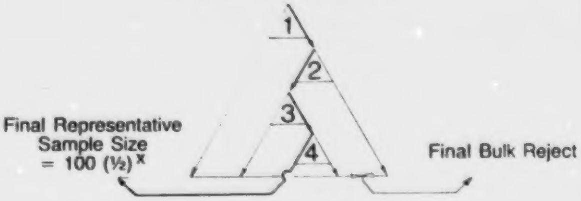


Table 1 Recommended Sample Splitter vs. Particle Size and Hopper Capacity

Maximum Particle Size		Recommended Minimum Slot Size		Riffler Hopper Size / Hopper Capacity(1), kg (lbs)					
mm	in.	mm	in.	0 / 0.17(0.4)	1 / 1.8(4)	2 / 3.5(7.5)	3 / 5.0(11.0)	4 / 7.0(15.0)	5 / 15.0(33.0)
11	7/16	25.4	1.0						SS-16-25
10	3/8	19.1	3/4						(SS-22-19) SS-16-25
8	5/16	16.0	5/8						(SS-22-19) SS-16-25
5	4 mesh	12.7	1/2			(SS-16-13)	(SS-24-13)	(SS-32-13)	SS-22-19 SS-16-25
4	5 mesh	9.5	3/8			(SS-22-10) SS-16-13	SS-24-13	SS-32-13	SS-22-19 SS-16-25
2	10 mesh	6.3	1/4		(SS-16-6)	(SS-32-6) SS-22-10 SS-16-13	SS-24-13	SS-64-6 SS-32-13	SS-22-19 SS-16-25
1	18 mesh	3.0	1/8	(SS-16-3) (Microsplitter)	SS-16-6	SS-32-6 SS-22-10 SS-16-13	SS-24-13	SS-64-6 SS-32-13	SS-22-19 SS-16-25
0.5	35 mesh	1.5	1/16	(SS-24-1.5) SS-16-3 (Microsplitter)	SS-16-6	SS-32-6 SS-22-10 SS-16-13	SS-24-13	SS-64-6 SS-32-13	SS-22-19 SS-16-25

Notes: (1) Based on 1602 kg/m^3 or 100 lbs./ft.^{3}, level filled hopper.

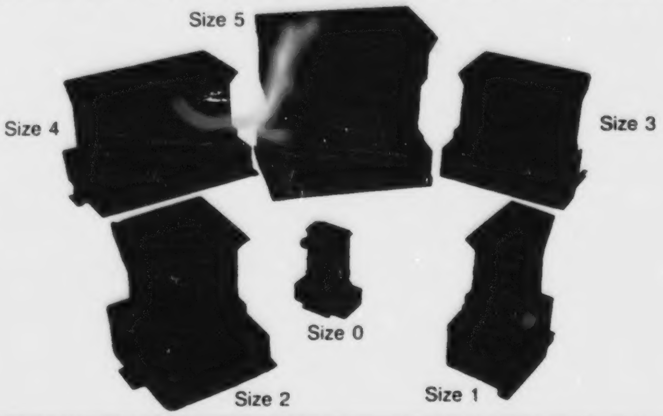
Table 2: Riffler Specifications

Model Number (5)	Total No. of Slots	Slot Size		Hopper Size	Hopper Volume (2)		Hopper Capacity (3)		Product Pan Volume (2)		Outside Dimensions						Approximate Shipping Weights (4)				Extra Pans		
											Length (A)		Width (B)		Height (C)		Net		Gross		Model	Gross Wts.	
		mm	in.		cc(1)	in.^{3}	kg	lbs	cc(1)	in.^{3}	mm	in.	mm	in.	mm	in.	kg	lbs	kg	lbs		kg	lbs
SS-24-1.5 (Microsplitter)	24	1.5	1/16	0	0.1	7	0.17	0.4	0.1	7	114	4.5	152	6	203	8	1.8	4.0	2.7	6.0	SS-0	0.2	0.5
SS-16-3 (Microsplitter)	16	3.0	1/8	0	0.1	7	0.17	0.4	0.1	7	114	4.5	152	6	203	8	1.8	4.0	2.7	6.0	SS-0	0.2	0.5
SS-16-6	16	6.0	1/4	1	1.1	67	1.8	4.0	1.1	67	178	7.0	279	11	305	12	3.6	8.0	5.4	12.0	SS-1	1.8	3.9
SS-32-6	32	6.0	1/4	2	2.1	130	3.5	7.5	2.4	148	305	12.0	279	11	305	12	5.0	11.0	7.3	16.0	SS-2	2.4	5.2
SS-64-6	64	6.0	1/4	4	4.2	256	7.0	15.0	4.9	300	508	20.0	279	11	305	12	11.4	25.0	15.9	35.0	SS-4	4.2	9.3
SS-22-10	22	10.0	3/8	2	2.1	130	3.5	7.5	2.4	148	305	12.0	279	11	305	12	5.4	12.0	6.8	15.0	SS-2	2.4	5.2
SS-16-13	16	13.0	1/2	2	2.1	130	3.5	7.5	2.4	148	305	12.0	279	11	305	12	5.0	11.0	6.8	15.0	SS-2	2.4	5.2
SS-24-13	24	13.0	1/2	3	3.1	190	5.0	11.0	3.8	233	381	15.0	279	11	305	12	9.1	20.0	13.2	29.0	SS-3X	4.1	9.0
SS-32-13	32	13.0	1/2	4	4.2	256	7.0	15.0	4.9	300	508	20.0	279	11	305	12	10.9	24.0	13.6	30.0	SS-4	4.2	9.3
SS-22-19	22	19.0	3/4	5	9.2	562	15.0	33.0	6.4	562	508	20.0	305	12	432	17	17.7	39.0	23.2	51.0	SS-5	4.5	10.0
SS-16-25	16	25.0	1.0	5	9.2	562	15.0	33.0	6.4	562	508	20.0	305	12	432	17	19.5	43.0	26.8	59.0	SS-5	4.5	10.0
All Stainless Construction																							
SS-24-1.5X (Microsplitter)	24	1.5	1/16	0	0.1	7	0.17	0.4	0.1	7	114	4.5	152	6	203	8	2.6	5.7	3.2	7.0	SS-0X	0.2	0.5
SS-16-3X (Microsplitter)	16	3.0	1/8	0	0.1	7	0.17	0.4	0.1	7	114	4.5	152	6	203	8	2.7	6.0	3.6	8.0	SS-0X	0.2	0.5
SS-16-6X	16	6.0	1/4	1	1.1	67	1.8	4.0	1.1	67	178	7.0	279	11	305	12	5.4	12.0	8.2	18.0	SS-1X	1.8	3.9
SS-16-13X	16	13.0	1/2	2	2.1	130	3.5	7.5	2.4	148	305	12.0	279	11	305	12	10.4	23.0	16.3	36.0	SS-2X	2.7	6.0
SS-24-13X	24	13.0	1/2	3	3.1	190	5.0	11.0	3.8	233	381	15.0	279	11	305	12	11.8	26,0	18.2	40.0	SS-3X	4.1	9.0
SS-32-13X	32	13.0	1/2	4	4.2	256	7.0	15.0	4.9	300	508	20.0	279	11	305	12	19.1	42.0	29.5	65.0	SS-4X	5.0	11.0

Notes:

(1) Multiply by 10^3
(2) Filled level
(3) Based on 1602 kg/m^2 or 100 lbs/ft.^{3} material.
(4) Includes "Sample Splitter" and two product pans.
(5) Pans designated with an "X" denote stainless steel. All other pans are aluminium alloy.
(6) Cleaning brush supplied with all models except for microsplitters.

Figure 3: Key below of Sample Splitters shown on front cover. These are identified by hopper size.



Recognized Leader In Dry Separation Technology Since 1948

Carpco, Inc.
4120 Haines Street
Jacksonville, Florida
U.S.A. 32206
904/353-3681
Telex. 5-6367

Represented by:

Electrostatic and Magnetic Separation Systems

SAMPLE SPLITTER INFORMATION

Sampling Procedure

1. Determine minimum final sample size for end use based on particle size, composition, liberation and specific gravity. Table 1 shows several examples for varying materials.
2. A representative portion of the parent lot of material to be tested must be obtained by an appropriate sampling procedure.
3. Use the correct sample splitter based on the quantity of material to be handled and particle size. Some materials such as coarse run-of-mine ores will necessitate large minimum sample quantities which will have to be reduced in particle size by crushing and/or grinding, and then riffled to obtain the desired representative end sample. Information on sample splitter selection is presented in Carpco Bulletin 8290.
4. Figure 1 illustrates a typical splitting or riffling procedure for a sample. Note that opposite cuts are taken to avoid possible splitting bias.
5. Clean the sample splitter with a pressure regulated air hose or by brushing between different samples to avoid contamination. Splitter models having hopper size 0 and 1 are fitted with hand-release dust shields to simplify clean-out.

CAUTION

WHEN WORKING WITH ANY DUSTY MATERIALS, CARE SHOULD BE TAKEN TO PROVIDE OPERATOR(S) OF LABORATORY OR INDUSTRIAL SEPARATING DEVICES WITH PROTECTIVE MASKS. PARTICULAR CARE SHOULD BE TAKEN WHEN THE MATERIALS ARE KNOWN TO BE HAZARDOUS OR TOXIC, I.E. POSITIVE DRAFT VENTILATION SHOULD BE CONSIDERED.

FIGURE 1: SAMPLE SPLITTING DIAGRAM

Total Sample = 100

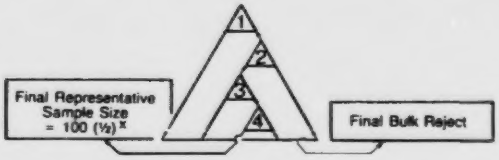


TABLE 1: EXAMPLES OF MINIMUM SAMPLE SIZE

Type of Material	Upper Limiting Screen Size of Material (90% Passing)			Recommended Minimum Sample Size	
	mm	inches	U.S. mesh	kg	lb.
1) Fertilizer Mixture **Description** 15 w/o P_2O_5, 15 w/o K_2O or 27.9 w/o or 30.5 v/o $(NH_4)_2HPO_4$ 48.4 w/o or 48.4 v/o $(NH_4)_2SO_4$ 23.7 w/o or 21.1 v/o KCL 100 w/o 100 v/o (SP. GR. of Mixture = 1.77) Type of Assay K_2O **Error** Permissible error due to sampling = 0.045 w/o with 1σ reliability	6 3 1	0.25 0.125 0.04	3 6 18	42.0 5.3 0.2	93 12 0.43
2) Porphyry Copper Ore **Description** 0.75 w/o Cu as chalcopyrite (2.17 w/o or 1.50 v/o chalcopyrite) SP. GR. of Ore = 2.90 Liberation at 65 mesh Type of Assay Cu **Error** Permissible error due to sampling = 0.01 w/o Cu or 0.015 w/o chalcopyrite with 1σ reliability	6 3 1 0.21	0.25 0.125 0.40 0.008	3 6 18 70	7.3 1.3 83×10^{-3} 1.7×10	16 3 0.2
3) Beach Sand Zircon Concentrate **Description** Rutile 0.3 Others 1.5 Zircon 98.2 Total 100 w/o (SP. GR. = 4.1) Type of Assay TiO_2 **Error** Permissible error due to sampling = 0.01 w/o or 0.01 v/o TiO_2 with 1σ reliability **Legend** w/o - Weight Percent v/o - Volume Percent	0.297 0.149	0.012 0.006	48 100	5.4×10^{-3} 0.68×10^{-3}	

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Jacksonville, Florida
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904/353-3681
Telex. 5-6367

Represented by:

Carpco, Inc.

PLS 1
Effective January, 1987

I. PRICE LIST FOR PRECISION SAMPLE SPLITTERS

SLOT SIZE	TOTAL NO. SLOTS	MODEL NO.	HOPPER SIZE	EX-WORKS PRICE
1/8" (3mm)	16	SS-16-3	0	$ 292.00
1/4" (6mm)	16	SS-16-6	1	$ 338.00
	32	SS-32-6	2	$ 455.00
	64	SS-64-6	4	$ 720.00
3/8" (10mm)	22	SS-22-10	2	DROPPED
1/2" (13mm)	16	SS-16-13	2	$ 398.00
	24	SS-24-13	3*	$ 548.00 ✓
	32	SS-32-13	4	$ 625.00
3/4" (19mm)	22	SS-22-19	5	$ 745.00
1" (25mm)	16	SS-16-25	5	$ 752.00 ✓

ALL-STAINLESS CONSTRUCTION MODELS for special applications

1/16" (1.5mm)	24	SS-24-1.5x	0x	$ 442.00
1/8" (3mm)	16	SS-16-3x	0x	$ 382.00
1/4" (6mm)	16	SS-16-6x	1x	$ 525.00
		SS-32-6x	2x	$ 685.00
1/2" (13mm)	16	SS-16-13x	2x	$ 645.00
	24	SS-24-13x	3x	$ 758.00
	32	SS-32-13x	4x	$ 860.00 ✓
1" (25mm)	16	SS-16-25x	5x	$1,124.00

PANS: TWO PRODUCT PANS ARE FURNISHED WITH EACH SPLITTER.

* Note: Due to its wide use in corrosive fertilizer sampling, Model SS-24-13 is always supplied with stainless steel pans.

EXTRA PAN SCHEDULE

Hopper Size	Price per Pan	Hopper Size	Price per Pan
0	$15.00 (aluminum)	0x	$18.00 (stainless)
1	$18.00 (aluminum)	1x	$26.00 (stainless)
2	$24.00 (aluminum)	2x	$32.00 (stainless)
4	$35.00 (aluminum)	3x	$44.00 (stainless)
5	$45.00 (aluminum)	4x	$58.00 (stainless)
		5x	$62.00 (stainless)

II. PRICE LIST FOR ALL OTHER SAMPLERS

	Price, Ex-Works
Model SP-410 Hand-Held Slurry Sampler........................	$185.00
Model SP-411 " " " "	$185.00
Model SP-412 " " " "	$215.00
Model SP-413 " " " "	$215.00
Model WS-220 Slurry Sampler................................	$3,810.00
Model DT-48X D-Tube Sampler................................	$1,395.00

PRICES AND SPECIFICATIONS SUBJECT TO CHANGE WITHOUT NOTICE.

APPENDIX IX

REPORT FROM MATRIX CONSULTANT LTD.

SAMPLING SYSTEMS
FOR POTASH
AT
NEPTUNE TERMINALS

Submitted to:

CANPOTEX Shipping Services Ltd.
North Vancouver, B.C.

Prepared by:

J.W. Merks
President
Matrix Consultants Limited
Vancouver, B.C.

August 30, 1985

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1. INTRODUCTION

At the request of Mr. S.A. Boskov, General Manger of CANPOTEX Shipping Services Ltd. a visit was paid to Neptune Bulk Terminals (Canada) Ltd. on August 20, 1985. The purpose of this visit was to evaluate design and operational parameters of the mechanical sampling systems for potash at this terminal, and to assess their potential to collect biased gross samples. The evaluation was based on the premise that gross samples should be unbiased for particle size distribution. For the percentage fine is an important quality characteristic in potash. The critical design and operational parameters for primary samplers and sample dividers, and their effect on accuracy and precision of gross samples of potash for size consist analysis, will be reviewed.

The history of these mechanical sampling systems was discussed with Mr. Brent S. Sands, Vice President, Operations with Neptune Bulk Terminals (Canada) Ltd., who was most co-operative during this visit to the site by making available appropriate drawings, and by assisting in the measurement of a number of parameters that were required to evaluate the sampling systems and to compile the report.

This report is divided into three (3) sections. In the second section the principles behind design and operation of primary samplers and sample dividers will be reviewed. Although critical parameters for mechanical sampling systems will be addressed briefly in this section, more detailed information on these subjects can be found in a textbook on "Sampling and Weighing of Bulk Solids", a copy of which was made available to CANPOTEX as a part of this report.

In the third section a number of basic conclusions and statements are presented. The current state of sampling technology would permit the design of effective mechanical sampling systems for most types of bulk solids. However, the systems at Neptune Terminals are poor examples of how to design a mechanical sampling system to determine the size consist analysis of potash.

- cont'd -

2. DISCUSSION

The design of these mechanical sampling systems and their mode of operation will be discussed in terms of the accuracy and precision characteristics of gross samples for the most relevant quality parameters of potash such as size consist analysis and grade. These mechanical sampling systems are identical in design so that the remarks in the following sections apply to both systems. At present only the system between the discharge system for rail cars and the storage shed is operational. The mechanical sampling system on top of the silo between the storage shed and the ship loader requires repairs. The question whether repairs to this system are justified depends largely on whether the operational system provides unbiased gross samples for the particle size distribution.

The components of a sampling system should be assessed on the basis of their suitability to collect and to process a gross sample for the particle size distribution. For a system that collects unbiased gross samples for size consist analysis, is unbiased for all other quality parameters. However, a system that collects an unbiased sample for the grade of a bulk solid such as potash, need not be unbiased for its particle size distribution.

Mechanical sampling systems should be checked for systematic errors upon completion of installation. In fact, the terms of the contract with the supplier of a sampling system should specify that the final payment is contingent upon the outcome of a bias check program. If the results prove that the sampling system is biased, the manufacturer should make modifications until it meets the requirements.

Vibrations during transportation and handling of potash may cause fines that result from abrasion between particles, to migrate. Hence, the finest size fraction in a consignment of potash is not expected to be distributed at random but would tend to occur in pockets that may show a Poisson distribution. This condition demands that a high frequency

- cont'd -

sampling regime be selected. At present the sampling interval for the primary cutter is about 4½ minutes so that a total of thirteen (13) increments per hour are collected. This regime is equivalent to one (1) primary increment from a mass of one-hundred (100) metric tons of potash, which should provide an acceptable degree of precision for most quality parameters. However, a sampling experiment would be required to determine the variability of the coarsest and finest size fractions so that the actual precision for this fraction of the size consist analysis can be measured in terms of a coefficient of variation.

2.1. Primary Sampler

This primary sampler is another example of a most unusual design which appears to be based on the premises that any small amount of material that is collected from a large particle flux at a transfer point, is a primary increment, and that any set of such increments constitutes an unbiased gross sample. For the design displays a complete disregard for accepted criteria for primary cutters so that the probability to collect, with this contraption, an unbiased gross sample for size consist analysis is very small indeed. It may, however, collect an unbiased gross sample for quality parameters such as sodium, potassium, or any others that are not significantly affected by a systematic error in the particle size distribution.

The primary sampler consists of six (6) sampling probes which are mounted onto a cylinder with a screw conveyor that transfers the sample flow to the secondary divider. The slot in each probe is 75 mm long and 15 mm wide, although some slots are deformed and smaller than 15 mm. These sampling probes are spaced at distances of 180 mm while the total width of the cutter is 900 mm. The mass of a primary increment is a function of the handling capacity, and generally ranges from 12 kg up to 15 kg.

- cont'd -

Between cuts the sampling probes, or the fingers on the primary cutter point upwards and are pressed against a baffle plate to prevent dust from entering the secondary divider between cuts, and moisture from running into the screw conveyor during cleaning.

During each cut the primary cutter is rotated into the trajectory of the potash flux from the main conveyor which causes some material to spill through the slots into the sampling probes. While returning to its stationary position the material in the sampling probes slides into the cylinder, and is then transferred into the Vezin divider.

A primary increment that is collected with this cutter, is an example of a partial stream cut from the upper section of the particle flux at the transfer point in the conveyor belt system. At the end of the conveyor a significant amount of the finest fraction in the potash stratum on the belt will have segregated to lower strata due to vibrations of the belt while it travels over the rollers. Hence, the upper stratum is expected to be deficient in fines so that a partial stream cut from the upper stratum only, will contain less fines than a full stream cut, and will thus be biased for its size consist analysis.

Critical design parameters for a linear sampler that is designed to collect a full stream cut for a primary increment from a particle flux at a transfer point, are the speed of the cutter, the width of its slot, and the position of the cutter edges in relation to the particle flux. Many variations of this time-tested design have been checked for systematic errors in mechanical sampling systems in different parts of the world, and were found to be unbiased for a wide range of bulk solids and handling capacities. For potash this type of primary sampler would have been an excellent choice.

In numerous national and international standards on sampling of bulk solids such as coal and iron ore, recommendations for the speed of a linear cutter generally range from 0.45 mps up to 0.6 mps while its width should not be less than three (3) times the top size of a bulk

- cont'd -

solid, or 10 mm for a top size of less than 3.3 mm. The plane of the cutter edges should be perpendicular to the particle flux at the point where the primary increment is collected.

For a critically designed linear cutter the mass of a primary increment is a function of the handling capacity only, and the next formula is used to calculate this sample mass:

$$\Delta m = \frac{M(c) * w}{3.6 * v(s)}$$

in which: Δm = mass of a primary increment in kg
$M(c)$ = handling capacity in mtph
$v(s)$ = speed of sampler in mps
w = width of sampler opening in m

The handling capacity for potash during discharge of rail cars ranges from 1,200 mtph up to 1,500 mtph. At this capacity a linear cutter that has a width of 15 mm, and that travels at a speed of 0.6 mps, collects a primary increment with a mass of only 5.6 kg. Yet, the mass of a partial stream cut with the primary cutter in this system ranges from 12 kg up to 15 kg. This mass was estimated by collecting a single increment in a PVC bag. Hence, a full stream primary increment that is collected with a critically designed linear sampler, would not only be unbiased but its mass would be even smaller than that collected with the present contraption.

Although the design of the primary cutter for this sampling system is expected to be biased, the device should be checked for systematic errors before further action is taken. Bias test programs are based on collecting a series of system samples with the primary cutter and a series of reference increments from the stopped conveyor belt, and on comparing measurements in these paired data sets. The actual number of paired data depends on the variability of the quality parameter of interest, on the bias detection limit for the test, and on the maximum permissible bias. In this case the bias test should be based on the

- cont'd -

particle size distribution, and in particular on the percentage undersize in the coarsest grade.

The design of a bias test program and the evaluation of bias test data requires experience and expertise in this field. The bias test should be properly designed and the required field work carried out with great care and under close supervision. The section on bias testing in the text on "Sampling and Weighing of Bulk Solids" should be consulted for more details on this subject.

2.2. Primary Sample Flow

Primary increments are discharged into a screw conveyor that transfers the sample flow to a Vezin divider. A screw conveyor is an effective device for transfer of dry and free-flowing bulk solids in low capacity applications. However, in a soft bulk solid such as potash the friction between particles during transfer causes size degradation in the sample flow, and results in anomalous amounts of fines. Some of these fines are expected to accumulate in the lowest section of the screw conveyor until the rotating spiral catches these fines, and transfers them into the Vezin divider. Hence, it is possible that fines from one gross sample are transferred into another.

This method of feeding the primary sample flow to the secondary stage, can only be changed if the current sampling device were replaced with a linear chute or bucket cutter. A decision to replace the present primary cutter, and thus to eliminate the need for a screw conveyor, is contingent upon the outcome of a bias test for this sampling system. For a screw conveyor is a poor choice if the percentage fines in a bulk solid with a narrow particle size range such as potash is an important quality parameter.

- cont'd -

2.3. Secondary Stage

The secondary stage of each system consists of a Vezin divider with a single sample collection slot. The opening of this slot is equal to approximately 1.25% of the surface of the circle that it describes, so that the division ratio for this divider is close to 1:80. Division ratios in excess of 1:50 rarely maintain a statistically significant correlation between measurements on primary increments and on corresponding sets of secondary increments. Yet it is imperative that such paired data sets be correlated. Otherwise a random number generator could be used to simulate data for large numbers of secondary increments on the basis of real measurements on a limited number of primary increments.

From each primary increment some seven (7) secondary increments are collected. A set of secondary increments was weighed, and their cumulative mass amounted to 175 grams so that the mass of a single secondary increment is only 25 grams. Based on a division ratio of 1:80, this mass of 0.175 kg for a set of secondary increments is equivalent to a mass of 14 kg for a primary increment which is within the expected range from 12 kg up to 15 kg. If coarse potash were sample manually, the mass of a primary increment should range from 0.15 kg up to 0.25 kg. Therefore the average mass of a secondary increment for size consist analysis should preferably be closer to 0.15 kg than to 0.025 kg.

The radii of the circles at the minimum and maximum width of the slot are approximately 0.10 m, and 0.22 m for circumferences of 0.62 m and 1.38 m. At the center of the particle flux this circumference is 1.0 m. The rotational speed of this Vezin divider turned out to be 30 rpm so that its tangential speed at the center of the primary sample flux is then: 1.0 * 30/60 = 0.5 mps. Hence, the speed of the slot at this point is below the critical speed of 0.6 mps so that the Vezin divider would collect unbiased secondary increments if its speed were the only criterion. The width of the slot at the center of the flux is about 12.5

- cont'd -

mm which exceeds the minimum width of three (3) times top size for coarse potash.

Only the division ratio of 1:80 is a cause for concern in this mechanical sampling system for potash. This type of Vezin divider would be more suited for this application if its division ratio were lower, and if the mass of the average secondary increment were closer to 0.1 kg. A lower division ratio would result in a larger but more precise gross sample. Its mass can be reduced by increasing the time interval between cuts which would result in a less precise gross sample. A more effective alternative to this Vezin divider would be a secondary system with two (2) continuous division stages.

- cont'd -

3. SUMMARY

Only the mechanical sampling system between the discharge facility for rail cars and the storage shed for potash at Neptune Bulk Terminals (Canada) Ltd. is functional. The sampling system between this shed and the ship loader requires extensive repairs. Each system consists of a primary cutter with an integrated screw conveyor that transfers the sample flow to a Vezin divider. Final system samples are collected in PVC bags that are tightly connected to the sample chute from the Vezin divider.

Neither the primary cutter nor the Vezin divider are suitable for this application. The design of this cutter defies all accepted criteria for primary samplers. For it collects partial stream cuts from a section in the particle flux at the transfer point, that is expected to be deficient in fines. The division ratio of 1:80 for the Vezin divider is much too high to do justice to the intrinsic precision of a gross sample that is collected at constant time intervals of 4½ minutes.

A bias test should be conducted to assess accuracy and precision characteristics for the primary sampler and the Vezin divider in the operational system. This test program should be based on a systematic comparison of measurements in reference increments, rejects from the Vezin divider, and final system samples. Reference increments should be collected from the stopped belt while each system increment should be collected from a position close to its paired reference increment. The rejects and the final system sample from each system increment should also be collected and kept in separate PVC bags. Between twenty (20) and thirty (30) measurements in three (3) sets of samples will be required to calculate the statistical parameters for this bias test, and to obtain an acceptable bias detection limit. In each set of samples undersize and oversize should be measured to assess the operation of this mechanical sampling system. More information on bias testing can be found in a text on "Sampling and Weighing of Bulk Solids".

- cont'd -

The outcome of this bias test will permit an effective course of action to be taken. Some alternatives to this cutter are linear chute or bucket cutters with overhead drives. A more efficient alternative to a Vezin divider with a single 1.25% slot is a rotary disc divider. The use of two (2) rotary disc dividers in series would not only permit division ratios between 1:100 up to 1:400 without a significant loss in precision, but also an increase in the number of primary increments, and thus a higher degree of precision for measurements on such gross samples.

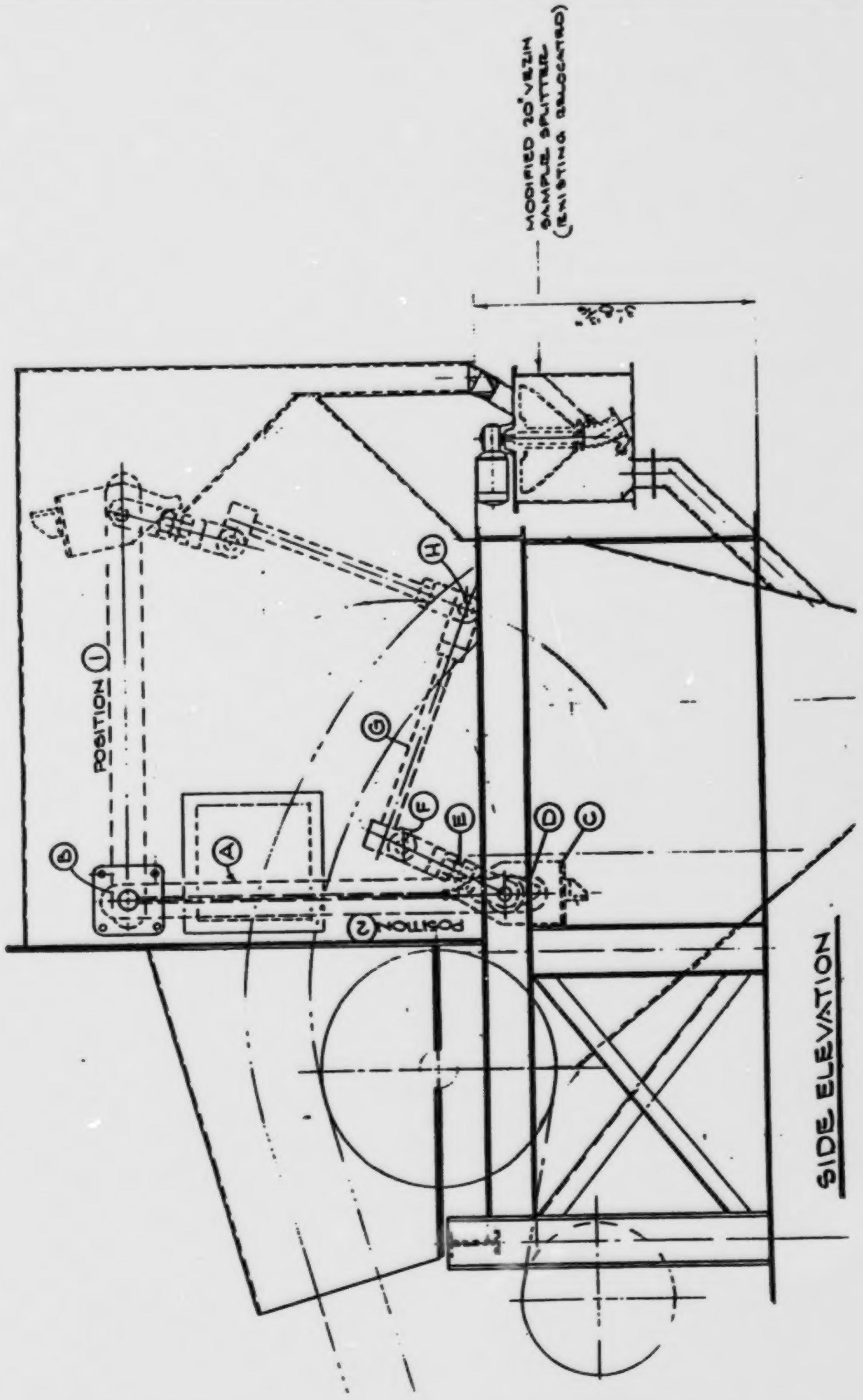
The percentage undersize in coarse potash is the most critical quality parameter for this grade so that a systematic error in this percentage would be a cause for concern and dispute between buyers and sellers. Efforts to closely monitor these amounts of fines in potash in rail cars and in consignments aboard vessels should be based on unbiased gross samples in which the most important particle size fractions can be measured with an acceptable degree of precision.

Submitted on : August 30, 1985

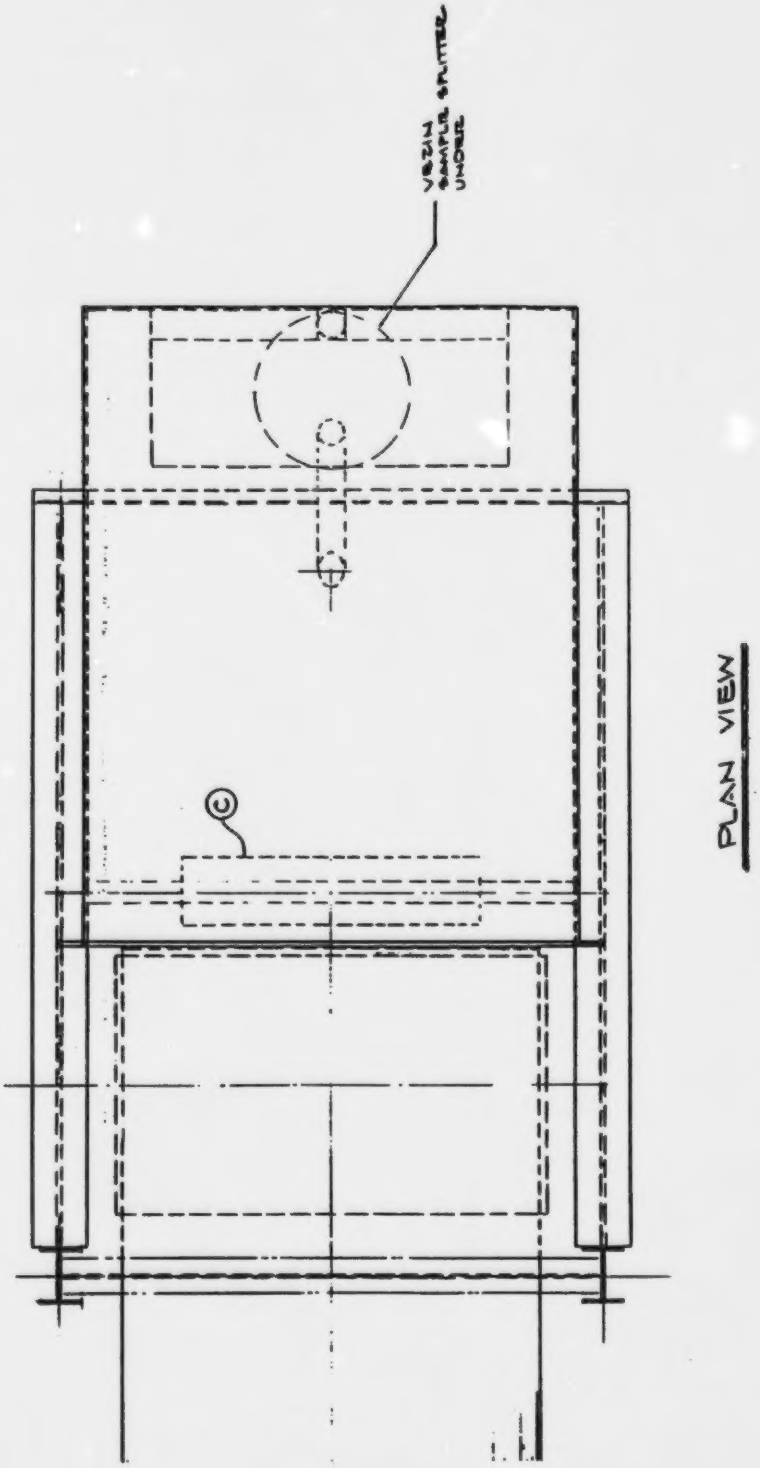
by : J.W. Merks
President
Matrix Consultants Limited

APPENDIX X

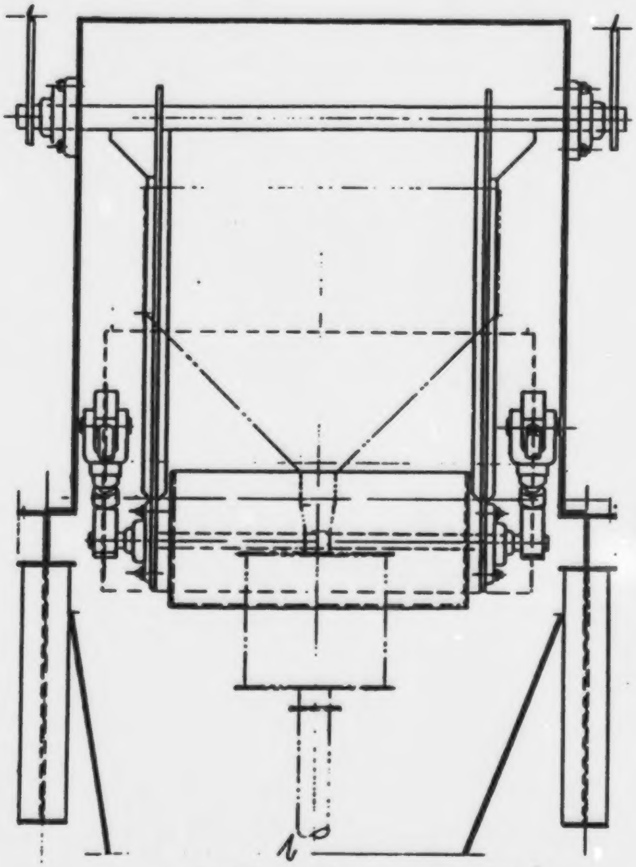
DRAWINGS OF KALIUM AUTOMATIC SAMPLER


MODIFIED 20" VEZIN
SAMPLE SPLITTER
(EXISTING RELOCATED)
3'-8⅝"
H
POSITION ①
G
F
E
D
C
B
A
POSITION ②
SIDE ELEVATION

JOB No.			**KALIUM CHEMICALS** **A DIVISION OF PPG CANADA INC.**
DRAWN	BC	18·NOV·87	**TITLE-** NEPTUNE TERMINALS
CHECKED			
APPRV'D			SWING ARM SAMPLER · SIDE ELEVATION
SCALE			**DRG. No.-** SK1·A **REV.** △


VEZIN
SAMPLE SPLITTER
UNDER
U
PLAN VIEW

JOB No.			**KALIUM CHEMICALS** A DIVISION OF PPG CANADA INC.
DRAWN	BC	18-NOV-87	
CHECKED			TITLE- NEPTUNE TERMINALS
APPRV'D			SWING ARM SAMPLER · PLAN VIEW
SCALE			DRG. No.- SK1-B REV.



END VIEW -(CUTAWAY)

JOB No.			**KALIUM CHEMICALS** **A DIVISION OF PPG CANADA INC.**
DRAWN	BC	18-NOV-87	
CHECKED			**TITLE-** NEPTUNE TERMINALS
APPRV'D			SWING ARM SAMPLER - END VIEW
SCALE			**DRG. No.-** SK1-C **REV.**

APPENDIX XI

SURVEY FORM FOR POTASH PLANTS SHIPMENT SAMPLING SYSTEM

SPPA Research & Development Committee
Offshore Product Quality - Product Degradation in Transit

Survey of Sampling and Analysis Performed by Saskatchewan Producers on Export Shipments

1. The following sampling and analyses are performed on this (or these) export products:

    1. Red Standard
    2. Pink Standard
    3. White Standard
    4. Fine Standard
    5. White Coarse
    6. Crystal Coarse
    7. Granular 270
    8. Granular 250
    9. Granular 265
    10. Superblend 210
    11. Superblend 300
    12. Industrial White Soluble 1.0%
    13. Industrial White Soluble 0.5%
    14. Semi-Industrial White Soluble
    15. Industrial White Granular
    16. Refined Chemical White Soluble
    17. Agricultural White
    18. Coarse

2. Do you use an automatic sampler? Yes No

    If yes, please briefly describe your automatic sampling system:

    ____

    ____

    ____

3. Do you take manual (grab) samples? Yes No

    If yes, please briefly describe the manual sampler which is used:

    ____

    ____

    ____

4. What is the sampling frequency? ____________
    (ie: # of increments or grabs per railcar)

5. What is the size of each increment or grab? _________lbs
    _________kg

6. What is the size of the primary sample? _________lbs
    (ie: weight of composite sample taken for each railcar) _________kg

7. What is the size of the final sample? _________lbs
    (ie: sample sent to the laboratory for analysis) _________kg

8. Is each railcar sampled? Yes No

If no, how many railcars? ______________

9. What type sample reduction device do you use in the Laboratory?

1. Coning and Quartering
2. Standard Riffle
3. Gated Riffle
4. Rotary Sample Divider

10. Which of the following analyses are performed on each railcar?

1. KCl
2. NaCl
3. Mg
4. Ca
5. Br
6. $S0_4$
7. Water Insolubles
8. Surface Moisture
9. Total Moisture
10. Anticaking Agent
11. Antidusting Agent
12. Amine
13. Particle Size Distribution
14. Other ____________

11. Which of the following analyses are performed on the composite (or lot) sample?

1. KCl
2. NaCl
3. Mg
4. Ca
5. Br
6. $S0_4$
7. Water Insolubles
8. Surface Moisture
9. Total Moisture
10. Anticaking Agent
11. Antidusting Agent
12. Amine
13. Particle Size Distribution
14. Other ____________

12. Miscellaneous Information:

Name: ____________________

Company: ____________________

Plant: ____________________

Signature: ____________________

APPENDIX XII

SAMPLING AND WEIGHING OF BULK SOLIDS
J. W. MERCKS

INCREMENTAL DIVISION AND MIXING BY CONING

pp: 263 - 268

[redacted]

Series on Bulk Materials Handling
Vol. 4 (1985)

SAMPLING AND WEIGHING OF BULK SOLIDS

J. W. Merks

First Edition 1985

TRANS TECH PUBLICATIONS

6.2.2 Incremental Division

Incremental division is a high precision procedure for dividing samples of fine and dry bulk solids. Incremental division which is sometimes called increment reduction, is an example of stratified systematic or stratified random sampling from two-dimensional populations of particles.

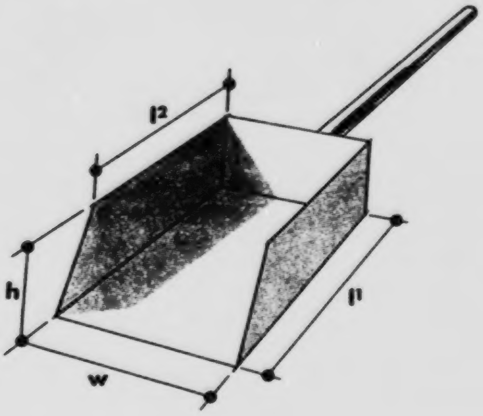
The process of incremental division combines logically with mixing by coning on stainless steel mixing trays of a special design so that a comminuted sample can be homogenized effectively to reduce the distribution heterogeneity prior to division. Mixing by coning with a set of trays is much more efficient and much less dusty than rolling or matting on a sheet of rubber or plastic.

Mixing by coning in combination with incremental division is a clean and precise procedure for the collection of a single subsample, or a set of subsamples, from a fine and dry sample. This efficient method is the most logical combination of mixing and division for a wide range of applications.

Incremental division, or increment reduction, is described in JIS M8100, a document published by the Japanese Standards Institute under the title "Common Rules for Methods of Sampling Bulk Materials of Mining Products". In this document the method of increment reduction is classified as a high precision procedure. Its main feature is that loss of dust during division of dried and pulverized samples is virtually eliminated so that the method is not only precise but also accurate.

Base metal smelters in particular have accepted incremental division as the standard procedure for division of dried samples of mineral concentrates. For in such concentrates the difference between the specific gravity of the valuable minerals and the siliceous material is high so that loss of dust during mixing and dividing with a riffle must logically result in higher metal assays in analysis fine samples on which final settlements are based. Buyers of valuable mineral concentrates are, obviously, not inclined to accept any procedure that is intrinsically biased, even though the systematic error is expected to be small, and therefore difficult to detect and expensive to prove.

In JIS M8100, incremental division and mass reduction with riffles are described as alternative procedures. Generally, the use of a riffle is limited to the division of samples that contain enough moisture to suppress loss of dust, but not quite enough moisture to clog the chutes of the riffle. These considerations lead to the conclusion that a riffle divider should not be used indiscriminately. Hence, for a wide range of bulk solids mixing by coning followed by incremental division is an accurate and precise

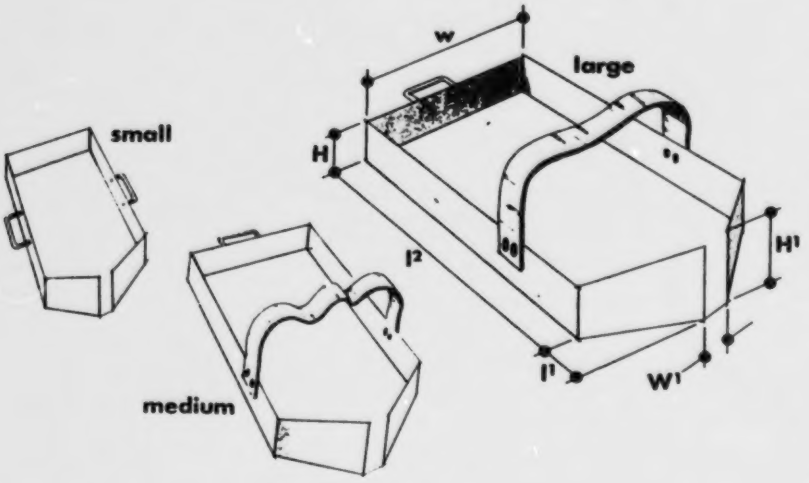


SCOOP NO.	TOPSIZE IN mm	VOLUME IN ml	DIMENSIONS IN mm				THICKNESS IN mm STAINLESS STEEL
			w	h	l1	l2	
1	1	15	30	15	30	25	.5
3	3	40	40	25	40	30	.5
5	5	75	50	30	50	40	1
10	10	125	60	35	60	50	1
15	15	200	70	40	70	60	2
20	20	300	80	45	80	70	2
30	30	400	90	50	90	80	2
40	40	800	110	65	110	95	2
50	50	1700	150	75	150	130	2

Fig. 6.12
Incremental Division Scoop

alternative to mixing and dividing with a riffle. In the case of fine and dry mineral concentrates, incremental division must be used exclusively.

Prior to increment reduction the sample should be carefully mixed by coning to reduce the distribution heterogeneity. Often, a manually collected gross sample is stratified to the extreme. Comminution tends to reduce the degree of segregation but a mixing step prior to division is the most effective measure to reduce the distribution variance, and thus, the variance component for the division step. Mixing by coning with stainless steel trays is a simple procedure to obtain homogeneous samples prior to



MEASURES IN MM	SMALL	MEDIUM	LARGE
L1	100	200	300
L2	200	400	600
W	150	250	400
W1	25	50	100
H	25	50	100
H1	50	100	150
Thickness	1	1.5	2

Fig. 6.13
Mixing Trays

incremental division. This procedure is particularly useful for preparing composites from a number of single samples.

In the next section a step-by-step description of mixing by coning with a set of stainless steel mixing trays is presented:

1.1 Select suitable pair of mixing trays to fit the sample mass that must be mixed by coning.

1.2 Mix by coning from one tray to the other while maintaining steady flow of material, and keeping the tip of the tray close to the top of the cone.

1.3 Upon completion of mixing, transfer the fines that adhere to the mixing tray, onto the top of the cone with a soft brush.

The number of times that a sample must be coned depends largely on the degree of segregation. Generally, ten (10) up to twenty-five (25) times will be sufficient to homogenize the material. Samples that are finer than 0.15 mm must be coned no less than twenty (20) times. The set of large mixing trays, and the set of medium size in the case of mineral concentrates, should be equipped with a leather strap at the front and a handle at the backside of each tray to facilitate the mixing process. Each small tray in a set should have a handle on its side so that material can be poured from one tray to the other conveniently and rapidly.

Although increment reduction is suited for the collection of a single subsample, the method is also convenient for sets of two (2) up to eight (8) subsamples. In the next section this division procedure is described:

2.1 Shake mixing tray with sample carefully to obtain flat stratum of uniform thickness.
2.2 Select increment scoop no. 3 for samples with a top size of 1 up to 2 mm, and increment scoop no. 1 for analysis fine samples.
2.3 Mark containers for subsamples with sequential number.
2.4 Select increment collection scheme from the following options:

For the actual collection of increments a stratified systematic and a stratified random regime can be used. In the case of a stratified systematic collection scheme each increment is collected from the center of a cell, but in a stratified random scheme each increment is collected at random within each cell. If a set of four (4) or more subsamples are required, the cells may become so small that the matter of systematic or random sampling becomes academic. In the next section the steps in a stratified systematic collection scheme are listed:

3.1 Select matrix for systematic collection of increments from the previous collection schemes.
3.2 Collect first increment from the center of the first cell in the matrix, and deposit in container no. 1.
3.3 Collect second increment from the center of the second cell in the matrix, and deposit in container no. 2.
3.4 Collect *n*th increment from the center of the *n*th cell in the matrix, and deposit in *n*th container.
3.5 Repeat steps 2, 3 and 4 until no less than eight (8) increments have been collected for each subsample.

Required Number of Subsamples

TWO

1	2	1
2	1	2
1	2	1
2	1	2
1	2	1
2	1	2

THREE

1	2	3	1
2	3	1	2
3	1	2	3
1	2	3	1
2	3	1	2
3	1	2	3

FOUR

1	2	3	4	1
2	3	4	1	2
3	4	1	2	3
4	1	2	3	4
1	2	3	4	1
2	3	4	1	2
3	4	1	2	3
4	1	2	3	4

FIVE

1	2	3	4
5	1	2	3
4	5	1	2
3	4	5	1
2	3	4	5
1	2	3	4
5	1	2	3
4	5	1	2
3	4	5	1
2	3	4	5

SIX

1	2	3	4	5
6	1	2	3	4
5	6	1	2	3
4	5	6	1	2
3	4	5	6	1
2	3	4	5	6
1	2	3	4	5
6	1	2	3	4
5	6	1	2	3
4	5	6	1	2
3	4	5	6	1
2	3	4	5	6

SEVEN

1	2	3	4	5	6	7	1
2	3	4	5	6	7	1	2
3	4	5	6	7	1	2	3
4	5	6	7	1	2	3	4
5	6	7	1	2	3	4	5
6	7	1	2	3	4	5	6
7	1	2	3	4	5	6	7

EIGHT

1	2	3	4	5	6	7	8	1
2	3	4	5	6	7	8	1	2
3	4	5	6	7	8	1	2	3
4	5	6	7	8	1	2	3	4
5	6	7	8	1	2	3	4	5
6	7	8	1	2	3	4	5	6
7	8	1	2	3	4	5	6	7
8	1	2	3	4	5	6	7	8

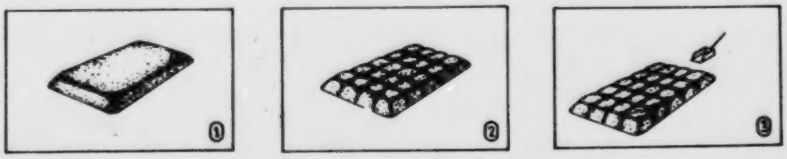


Fig. 6.14
Incremental Division

Since stratified random sampling is based on the collection of increments at random within each cell, this scheme is more suitable for a limited number of subsamples. In the next section a collection scheme for a single subsample is described. In the case that a set of two (2) up to four (4) subsamples is required, a suitable matrix must be selected from the previous series:

4.1 Mark a 4 x 5 matrix on the particle stratum in the mixing tray.
4.2 Collect first increment at random within the first cell, and deposit in sample container.
4.3 Collect second increment at random within the second cell, and deposit.
4.4 Collect *n*th increment at random within the *n*th cell, and deposit.
4.5 Repeat until twenty (20) increments are collected and deposited in the container for a subsample.

Generally, a subsample should consist of no less than eight (8) increments. Each increment must be collected by inserting the scoop under an angle of 30 to 45 degrees until it touches the mixing tray, and by pushing it forward with a quick motion to fill it while the handle of the scoop is being moved in downward direction. A baffle plate that is slightly larger than the opening of the scoop facilitates the collection of increments. A particle stratum on a mixing tray must not be thicker than the effective length of the scoop so that each increment will be collected from the entire thickness of the stratum. Hence, the process of incremental division is a small scale model of a manual sampling regime for a two-dimensional sampling unit.

A typical application for systematic increment reduction is to divide an analysis fine sample of mineral concentrates into a number of identical subsamples for submission to different laboratories. Random selection of increments from each cell in a matrix is more suited to collect a subsample from airdried or dried samples, or to collect a subsample from a composite sample that consists of two or more single samples.

6.3 Screening

Earlier in this chapter it was implied that screening should be avoided if at all possible. If the sample contains native metals, a sample mass of up to 2 kg must often be pulverized to obtain an acceptable degree of precision. Such large samples are alternately pulverized and screened to remove

APPENDIX XIII

PAPERS ON THE EVALUATION OF VARIOUS SAMPLE REDUCTION EQUIPMENT

X 453

Evaluation of Two-Way Riffles for Precise Division of Blended Fertilizers

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Evaluation of two-way riffles showed that gated riffles are superior to open-chute riffles. The proper technique appears to play a significant role in subdivision of blended fertilizers with the open-chute riffle, while the use of the gated riffle virtually eliminates error due to technique. A blended fertilizer prepared from materials having disparate particle sizes can be divided with a gated riffle to give a subsample that is representative of the original lot.

There has been considerable discussion of the contribution that dividing an unground sample makes to the total error of the chemical analysis of a fertilizer. There is a consensus that a blended fertilizer containing materials that are of significantly different particle size is difficult to divide into subsamples representative of the original lot. Other factors that contribute to the total error of the analysis have been studied extensively; sample reduction has received only minor attention.

Miles and Quackenbush (1) developed estimations for the magnitude of the contribution of sample reduction to total chemical analysis. They recommended reduction of composite samples to laboratory samples by using a two-way riffle rather than by hand (cone and quartering technique) because the riffle-reduced samples were less variable. Their work also showed that the variation associated with riffle reduction was about the same as that contributed by the analytical methods. Conversely, a Florida sampling study (2) recently concluded that in the case of the belt discharge samples, 90% of what is called total variance is due to riffling.

The purpose of this study was to examine the variations associated with two-way riffles of different sizes and dimensions on the division of blended fertilizers. Laboratory blends were prepared from closely matched materials, materials of specific screen fractions that were significantly divergent, and materials that are available in the market place. Results from weighing divided samples and from chemical analyses were statistically evaluated.

Experimental

Five riffles were evaluated during the study. Three of the riffles were manufactured by the same company and are designated A, B, and C. One riffle was manufactured by another company and is designated small riffle. The fifth riffle was constructed by the Tennessee Valley Authority (TVA) and is designated large riffle. All riffles are of similar design except that riffles A, B, and C have a hopper with a bottom discharge slot and a cutoff gate, and are called gated riffles. The dimensions of the riffles are shown in Table 1.

Procedure

Gated Riffles

Pour mixed samples ($\leq$1800 g) into the hopper, allowing formation of a natural cone or ridge. Without leveling or further disturbing the coned or ridged material, and with 2 pans in place below the riffle, completely open the gate with one motion. Allow the entire sample to flow through the riffle into the pans. If caked material bridges across the chute opening, force it through to the pans, combine, and reriffle.

Open-Chute Riffles

Mix the sample, transfer to the delivery pan, and level by hand. With 2 pans below the riffle, pour the material from the delivery pan in an even flow from ca 75 mm above the chutes in such a manner that the sheet of falling particles strikes near the center of the chutes. The sheet of falling material should extend from one end of the chute compartment to the other so that approximately the same amount of sample falls into each chute. *Do not* use a delivery pan longer than the chute compartment and *do not* pour sample from the end or corner of the delivery pan.

F. J. Johnson is Associate Referee on Phosphorus.

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Table 1. Riffle dimensions

Riffle	No. of chutes	Chute width, mm	Hopper size		Height, cm
			Top	Bottom	
A	16	13	18 x 20 cm	13 mm x 20 cm	30
B	22	10	18 x 20 cm	13 mm x 20 cm	30
C	32	6	18 x 20 cm	13 mm x 20 cm	30
Small	8	13	10 x 15 cm	38 mm x 10 cm	17
Large	18	13	13 x 30 cm	44 mm x 28 cm	37

Results and Discussion

A theoretical experiment was designed to determine the extent of subsample weight variation caused by dividing with different riffles. A mixture of 915 g 3 mm glass beads and 915 g 1 mm glass beads was divided by each of the 5 riffles according to the outline in Fig. 1. Subsamples 3 through 10 were weighed and the results are shown in Table 2. The data show that use of riffle A produced the smallest variance and range. Since subsamples 3, 4, 5, and 6 should represent equal portions of 915 g of sample, a percentage of the material can be calculated for each subsample. Likewise subsamples 7, 8, 9, and 10 should represent equal portions of 458 g of sample and the same calculation can be performed. Variances calculated for each riffle showed that riffles A and B were statistically better than the other 3 riffles. These data are shown in Table 3. Due to the similarity of the design of riffles A and B and since the results from this experiment were equivalent, riffle B was not used in further tests. Results from using riffle C were significantly different than those from riffle A and the chutes in riffle C are not 2.5 times as large as the largest fertilizer particle (a rule followed by many riffle manufacturers), therefore, its use was also discontinued.

Subsamples 5–10 from the glass bead experiment were screened and weighed, and the results are shown in Table 4. The data show that the variances for the small and large riffles are significantly larger than the variance for riffle A.

To avert the argument that subdivision of glass beads has nothing to do with fertilizers made up of disparate particles, another theoretical experiment was performed using fertilizer materials. Diammonium phosphate (DAP) and potassium chloride (KCl) were used in the study. Both materials were screened, and 820 g of a minus 6- plus 8-mesh fraction of DAP was mixed with an equal portion of minus 14- plus

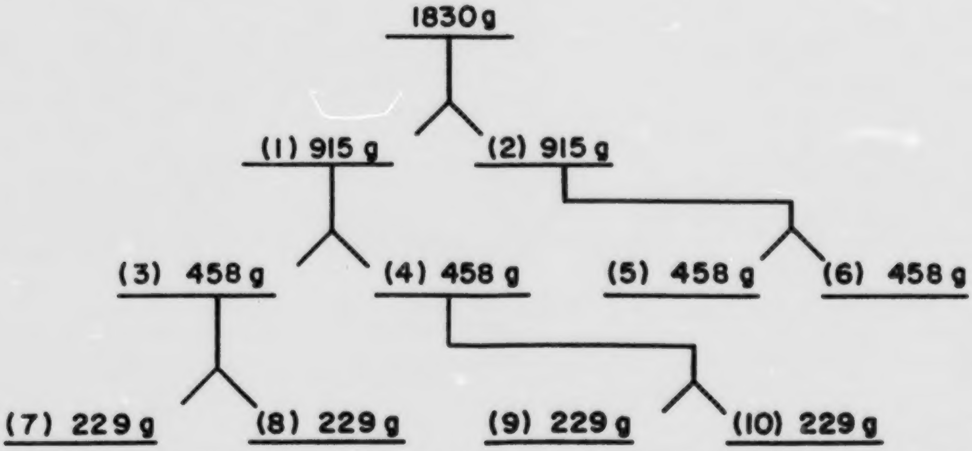

FIG. 1—Outline for division of sample to obtain 10 subsamples. Numbers in parentheses denote subsample numbers.

Table 2. Weight (g) comparison for the subdivision of glass beads

	Riffle				
Subsample No.	A	B	C	Small	Large
3	456.6	454.8	451.6	545.9	438.6
4	457.6	450.4	454.5	433.0	442.5
5	457.1	460.5	456.3	389.5	449.2
6	455.1	457.8	432.6	406.4	453.0
Av.	456.6	455.9	448.8	443.7	445.8
Variance	1.1667	18.743	119.67	4963	42.04
Range	2.5	10.1	23.7	156.4	14.4
7	229.4	227.8	227.6	255.9	213.3
8	226.6	226.2	222.7	288.4	221.0
9	229.5	225.8	226.9	216.5	216.7
10	227.5	222.8	226.5	214.2	223.7
Av.	228.3	225.7	225.9	243.8	218.7
Variance	2.0567	4.3567	4.8292	1252	21.15
Range	2.9	5.0	4.9	74.2	10.4

Table 3. Comparison of division ratios (weight %) of glass bead subsamples after riffle division

	Riffle				
Subsample No.	A	B	C	Small	Large
3	49.95	50.02	49.84	55.77	49.78
4	50.05	49.98	50.16	44.23	50.22
5	50.11	50.15	51.33	48.94	49.79
6	49.89	49.85	48.67	51.06	50.21
7	50.31	50.18	50.54	47.01	49.08
8	49.69	49.82	49.46	52.99	50.92
9	50.22	50.33	50.04	50.27	49.21
10	49.78	49.67	49.96	49.73	50.79
Variance	0.04546	0.04691	0.5965	12.4083	0.4466
F ratio	—	1.03	13.12	273	9.82

Table 4. Comparison of composition (weight %) of subsamples of glass beads as determined by sieve analysis

	Riffle					
	A		Small		Large	
Subsample No.	3 mm	1 mm	3 mm	1 mm	3 mm	1 mm
5	50.23	49.77	47.21	52.79	51.56	48.44
6	50.33	49.67	49.64	50.36	50.18	49.82
7	50.33	49.67	50.49	49.51	52.02	47.98
8	50.24	49.76	50.88	49.12	49.43	50.57
9	49.98	50.02	55.77	44.23	51.64	48.36
10	49.49	50.51	54.65	45.35	48.03	51.97
Variance	0.10576		10.2804		2.4157	
F ratio	—		97.2		22.8	

16-mesh KCl. Riffle A and the 2 open-chute riffles (large and small) were used to subdivide the blend according to the outline in Fig. 2. Each subsample was screened and weighed, and percentage of material in each screen fraction calculated. This procedure was repeated 5 times for each riffle; the data are shown in Table 5. Again, the 2 open-chute riffle variances are significantly larger than the variance for riffle A.

From the DAP-KCl study, each subsample from replicate No. 5 of riffle A was recombined, mixed, prepared, and analyzed for nutrient content; the results of the chemical analysis as well as the calculated grade from the sieve analysis

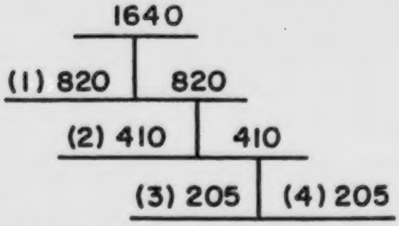

FIG. 2—Outline for division of DAP-KCl blend to obtain 4 subsamples. Numbers in parentheses denote subsample numbers.

for each subsample are shown in Table 6. From these data a total variance for N, P_2O_5, and K_2O can be calculated from the chemical analysis which includes riffling, sample preparation, mixing, and analytical methods. The variances for N, P_2O_5, and K_2O from the calculated grades are strictly due to riffling. These variances are also shown in Table 6. The variance due to riffling with riffle A is less than the analytical variance for N and P_2O_5, but greater than for K_2O.

Table 5. Composition (weight %) of subsamples of DAP-KCl blends with disparate particle sizes

Replicate No.	Subsample No.	Riffle					
		A		Small		Large	
		DAP	KCl	DAP	KCl	DAP	KCl
1	1	49.89	50.11	47.51	52.49	49.94	50.06
	2	49.91	50.09	54.35	45.65	51.14	48.86
	3	50.32	49.68	51.08	48.92	50.42	49.58
	4	49.71	50.29	50.35	49.65	47.90	52.10
2	1	50.01	49.99	48.83	51.17	50.59	49.41
	2	50.17	49.83	50.37	49.63	51.18	48.82
	3	49.47	50.53	50.43	49.57	50.35	49.65
	4	49.70	50.30	53.32	46.68	49.53	50.47
3	1	50.26	49.74	48.91	51.09	49.84	50.16
	2	49.51	50.49	51.66	48.34	49.81	50.19
	3	49.90	50.10	49.20	50.80	50.40	49.60
	4	49.30	50.70	51.75	48.25	50.45	49.55
4	1	50.07	49.93	49.36	50.64	50.40	49.60
	2	50.36	49.64	52.83	47.17	50.33	49.67
	3	49.66	50.34	50.36	49.64	47.21	52.79
	4	49.61	50.39	47.11	52.89	50.02	49.98
5	1	49.74	50.26	48.90	51.10	50.15	49.85
	2	49.90	50.10	53.10	46.90	49.72	50.28
	3	50.07	49.93	48.30	51.70	50.10	49.90
	4	50.68	49.32	48.90	51.10	49.14	50.86
Error mean square[a]		0.12819		4.863		1.0153	
F ratio		—		37.9		7.92	

[a] Error mean square = pooled variance of 5 sets of data.

Table 6. Comparison of calculated grade from sieve analysis with chemical analysis of the DAP-KCl blend after subdivision with riffle A

Subsample No.	Weight per cent					
	Calculated grade			Chemical analysis		
	N	P_2O_5	K_2O	N	P_2O_5	K_2O
1	8.95	23.34	29.64	8.92	23.44	29.95
2	8.98	23.41	29.54	9.04	23.79	29.49
3	9.01	23.49	29.44	9.10	23.78	29.40
4	9.10	23.78	29.08	9.25	24.18	29.27
Riffle variance	0.0055	0.0374	0.0596	—	—	—
Total variance	—	—	—	0.0188	0.0915	0.0875
Analytical variance[a]	—	—	—	0.0133	0.0541	0.0279

[a] Total variance minus riffle variance.

Four materials, DAP, triple superphosphate (TSP), ammonium sulfate (Am Sul), and KCl, were selected to prepare blends as they might occur in a fertilizer plant. The materials were screened and the sieve analysis data are shown in Table 7. Four blends, each weighing 1830 g, were prepared in triplicate for subdivision with riffle A and the 2 open-chute riffles. All 4 materials were used to prepare a 13-13-13 grade. Three materials—Am Sul, DAP, and KCl—were used to prepare a 15-15-15 grade. A 15-15-0 grade was prepared from DAP, TSP, and Am Sul; a 9-23-30 grade was prepared from DAP and KCl. Each blend was subdivided according to the outline in Fig. 1. The No. 10 subsample from each lote was prepared and analyzed 5 times for nutrient content. The data are shown in Table 8 and are compared to the calculated value. The Student *t*-test shows that only 2 results for riffle A, 2 results for the small, and 3 results for the large riffles are significantly different from the calculated values. These results show that there is no significant difference among the riffles tested, if reasonably matched materials and proper technique are used.

Table 7. Sieve analysis of materials used in simulated blends

Material	Weight, %					
	+6	+8	+12	+14	+16	Pan
Am Sul	0.17	8.14	66.78	19.92	3.75	1.24
DAP	1.16	23.25	51.11	13.76	4.78	5.94
TSP	5.44	60.28	33.15	1.05	0.04	0.04
KCl	0.81	19.07	43.12	22.71	7.55	6.75

Table 8. Comparison of chemical analysis of blends with calculated grades

Nutrient	Calculated, %	Riffle[a]		
		A	Small	Large
N	13.15	13.19	13.28	13.06
P_2O_5	13.16	13.02	13.04	13.79[b]
K_2O	13.16	13.35	13.18	13.27
N	14.89	15.06	14.91	14.91
P_2O_5	14.90	15.33[b]	14.61	14.88
K_2O	14.87	14.97	14.99	15.03
N	15.06	15.06	15.26	15.01
P_2O_5	15.04	15.28	15.28[b]	14.78
N	9.08	9.38[b]	9.34[b]	9.23[b]
P_2O_5	23.09	23.45	23.07	23.31
K_2O	30.08	29.97	29.91	31.27[b]

[a] Each value is the average (%) of 5 determinations.

[b] Significantly different at $P(t) = 0.05$ (df = 4).

Table 9. Sieve analysis of industry materials

Material	Weight, %					
	+6	+8	+12	+14	+16	Pan
Am Sul	1.13	6.73	32.80	21.24	11.45	26.66
DAP	0.57	22.29	62.00	9.96	2.82	2.37
TSP	7.29	59.99	27.91	3.11	0.96	0.74
KCl	3.01	13.12	28.96	23.11	10.92	20.89

Table 10. Comparison of chemical analysis of blend with calculated grade using riffle A

Subsample No.	Chemical analysis, %		
	N	P_2O_5	K_2O
7	13.35	13.14	13.22
8	13.25	13.49	13.10
9	13.33	13.25	13.18
10	13.30	13.31	13.14
Av.	13.31	13.30	13.16
Variance	0.00189	0.02143	0.00267
Calculated	13.33	13.21	13.19

To determine if riffle A would precisely divide blends made from materials with widely varying particle sizes, 4 fertilizer materials were obtained from an industry source. The screen data for the materials are shown in Table 9. A 13-13-13 grade blended fertilizer was prepared using all of the materials and subdivided according to the outline in Fig. 1. Subsamples 7–10 were each analyzed and compared to the calculated grade. These data are shown in Table 10. Each subsample shows good agreement with the calculated grade.

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(1) Miles, S. R., & Quackenbush, F. W. (1955) *J. Assoc. Off. Agric. Chem.* 38, 108–130

(2) "A Study of Official Sampling Techniques for Bulk Fertilizer in the State of Florida" (August 1976) prepared for Florida Agricultural Research Institute by Arthur D. Little, Inc., Acorn Park, Cambridge, MA 02140

This paper was presented at the 92nd Annual Meeting of the AOAC, Oct. 16–19, 1978, at Washington, DC.

APPENDIX XIV

COAL SAMPLING PROCEDURE

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OPERATING INSTRUCTIONS

FOR

SAMPLING SYSTEMS

BERTH 1

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MECHANICAL SAMPLING SYSTEM AT BERTH 1

OPERATING INSTRUCTIONS

1.0. START-UP PROCEDURE

1.1. Reset switches for "METRIC TONNES PER PRIMARY CUT" to zero.

1.2. Set required "METRIC TONNES PER PRIMARY CUT" on this set of switches in accordance with the sampling regime for a particular consignment of coal. (see Sampling Data Form)

Example : If the calculated "METRIC TONNES PER PRIMARY CUT" setting is 138 mt, then the switches are set to a value of 14.

1.3. Reset switches for "METRIC TONNES PER LOT" to zero.

1.4. Set required "METRIC TONNES PER LOT" on this set of switches in accordance with the sampling regime for a particular consignment of coal. (see Sampling Data Form)

Example : If the required "METRIC TONNES PER LOT" setting is 4,500 mt, then the switches are set to a value of 450.

1.5. The green light on the "SAMPLING PLANT MASTER CONTROL" indicates that the mechanical sampling system is ready for operation.

* * * * *

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2.0. OPERATING PROCEDURES

2.1. Push "SYSTEM START" button on the "SAMPLING PLANT MASTER CONTROL" to activate the mechanical sampling system.

Remark 1 : This "SYSTEM START" button controls hydraulic pumps for sample cutters in the secondary system, the hammer mill for the assay sample flow, and the roll crusher for the moisture sample flow.

Remark 2 : If Conveyor 5A to the shiploader at Berth 1 is running, the following conveyors in the secondary system are operational:

Primary Sample Flow

Conveyor 2SP6 : from Holding Hopper to Duplex Cutter
Conveyor 2SP12 : Reject Return Conveyor to Conveyor 5A

Assay Sample Flow

Conveyor 2SP7 : from Duplex Cutter to Hammer Mill
Conveyor 2SP8 : from Hammer Mill to Stage III Cutter
Conveyor 2SP9 : from Stage III Cutter to Stage IV Cutter

Moisture Sample Flow

Conveyor 2SP10 : from Duplex Cutter to Stage III Cutter
Conveyor 2SP11 : from Roll Crusher to Stage IV Cutter

Remark 3 : If Conveyor 4B to the surge hopper is running, the following conveyors for the primary sample flow are operational:

Conveyor 2SP1	from Primary Sampler East to Conveyor 2SP5
Conveyor 2SP2	from Primary Sampler West to Conveyor 2SP5
Conveyor 2SP5	Primary Sample Flow to Holding Hopper

* * * * *

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2.0. OPERATING PROCEDURES (cont'd)

2.2. Set "PRIMARY CUTTER" selector switch to "AUTO" mode.

Remark 1 : A green indicator light will come on to signify that the primary cutter is in automatic mode of operation.

Remark 2 : The primary cutter is activated by a signal from the belt scale for Conveyor 4B as soon as the amount of wet metric tonnes that is preset on "METRIC TONNES PER PRIMARY CUT" switches, has passed over this scale.

Remark 3 : As soon as amount of wet metric tonnes that passed over belt scale, is equal to the value set on "METRIC TONNES PER LOT" switches, this counter will automatically reset to zero. The "LOT COMPLETE" light on "SAMPLE LOT" will come on, and an audible signal will sound in the control room, at the sampling plant and at the sample preparation facility. At this time the sampler must initiate the secondary cycle by pushing "CYCLE START" so that the remaining primary increments for the lot are processed.

Remark 4 : The actual number of primary increments for each lot must be recorded on the corresponding Data Sheet, and the counter should be reset to zero after a lot is finished.

* * * * *

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3.0. OPERATIONS COMPLETED

3.1. Set "PRIMARY CUTTER" selector switch to "OFF" mode.

Remark 1 : This "OFF" position must also be selected in the case that the control light for the primary cutter indicates an error condition that requires intervention (see System Failures).

Remark 2 : Since the wet mass of the last lot in a consignment is not likely to be equal to the value on the "METRIC TONNES PER LOT" counter, the last cycle for the final lot must be activated manually by pushing the "CYCLE START" button.

* * * * *

4.0. PROCESSING OF SAMPLES

4.1. Upon completion of the last secondary cycle for a lot remove gross samples for total moisture and for other quality parameters from the corresponding sample containers, and process each set in accordance with applicable instructions.

Remark 1 : Gross samples for assay and moisture must be removed from the containers immediately following the completion of the last cycle for a lot. Each sample is first mixed by pouring from one PVC container into another no less than four (4) times and then divided with the Gilson SP1 divider until a subsample of the required mass is obtained. Next, each set of samples (assay and moisture) is either submitted to the laboratory for processing and testing, or taken to the preparation plant for air drying and drying.

* * *

- cont'd -

5.0. SWITCHING BETWEEN GRADES

5.1. Collect no less than three (3) primary increments in rapid succession by activating the primary cutter manually as soon the loading of another grade or type of coal, either into the surge bin or into the ship, has started.

5.2. Push "CYCLE START" to activate secondary system manually, and to purge it with the coal that is being loaded.

5.3. Upon completion of secondary cycle remove all material from sample containers, and ensure that containers for assay and moisture samples are both empty and clean.

5.4. Inspect sampling system for residues of wet coal from the previous consignment.

Remark 1 : If the time interval between the next loading of a ship and the completion of washing and cleaning of the sampling system with water is less than four (4) hours, the secondary system must not be washed with water. In that case the system must be cleaned dry by processing through the secondary system three (3) up to four (4) primary increments that are collected by manually activating the primary cutter. Upon completion sample containers for moisture and quality should be empty.

Remark 2 : Conveyors and feeders should not only be running while the secondary system is being washed and cleaned with water, but also upon completion to ensure rapid drying.

Remark 3 : During freezing weather the sampling system must not be cleaned with water to avoid ice formation within the system. Under those conditions procedures described in Section 7.0 - Winterization should be applied.

* * * * *

- cont'd -

6.0. SYSTEM FAILURES

6.1. General

The control panel for the sampling system at Berth 1 shows a large number of fault conditions for individual components. Some types of faults also trigger an audible alarm to alert the sampler that his intervention is required. Most failures require the services of maintenance. If the system fails completely, primary increments must be collected manually (hand sampling) at the transfer points in the conveyor belts under the large surge bin. If both conveyors are running, primary increments should alternately be collected from each transfer point. If the secondary system fails, but if the primary cutter functions properly, a gross sample can be collected manually from the reject return conveyor (semi automatic).

6.2. Primary sampler

At times the primary sampler may not trigger the end switch which results in a fault condition and an audible alarm on the control panel. This error condition can often be corrected by setting the "PRIMARY CUTTER" selector switch to the "OFF" mode, and by then jogging the primary sampler manually. The device should first be switched to manual control by setting the selector switch for the primary sampler to the "MANUAL" mode. This switch is located near the cable drive on the upper floor of the sampling system. Next, the primary sampler is jogged by pushing the button on this switch to move the sampler a small distance and then back in order to activate the end switch and correct this error condition. The switch should be returned to "AUTO" mode before returning to the control room. The sampling system cannot be reactivated if this switch is left in the "MANUAL" mode of operation. At times problems with the primary cutter can be solved by removing the fine coal that is compacted on the support rail of the primary sampler near the end switch. This coal should not be removed with water for it may leak onto conveyor belts 2SP1 and 2SP2.

- cont'd -

In the case that coal is simultaneously reclaimed from the stockpile and dumped from unit trains the handling capacity often exceeds 10,000 mtph. Although the mass interval between increments is fixed and preset, the time interval between primary increments depends on the actual loading capacity. At 10,000 mtph this time interval is too short for a primary increment to discharge completely from the bucket cutter. In this case the "METRIC TONNES PER PRIMARY CUT" switch should be adjusted to no less than "30" which is equivalent to 300 metric ton per primary increment.

6.3. Conveyors to Holding Hopper

Shuttle conveyors 2SP1 & 2SP2 are equipped with limit speed controls that can be adjusted in the control room. These controls should be set between 60% and 75% to avoid errors conditions for the sampling system. If these controls are set too high error messages on the control panel will light up and the alarm will sound.

6.4. Sampler Cutters and Feeders

Check all cutters and feeders regularly, and preferably during secondary cycles, to ensure that they function properly and that no plug-ups occur.

6.5. Roll Crusher for Moisture Sample

The belt drive of the crusher for the moisture sample may break and cause a fault condition for the secondary system. Replacement of this belt is a simple matter so that an effort should be made to have maintenance attend to its replacement as soon as possible. If the crusher fails to start, check for lumps of coal between the rolls, and remove with a piece of wood.

- cont'd -

6.6. Hydraulic Pumps

All cutters in the secondary sampling system operate hydraulically. Check hydraulic fluid level at the start of each shift to ensure that the cutters will function properly.

* * * * *

7.0. WINTERIZATION

7.1. Under freezing winter conditions conveyor belts and feeders should be run for at least four (4) hours during each shift even if no coal is being loaded. This procedure ensures that all belts and feeders are operational and at working temperature when loading commences.

7.2. Before the loading of a vessel commences all belts and feeders in the sampling system should be started to ensure that these components warm up to working temperature. Next, hoppers and chutes should be checked for frozen coal, and cleaned without using water if necessary.

7.3. As soon as loading has started a set of three (3) up to four (4) primary increments should be collected and processed through the secondary system. Read also Section 6.0. - "Switching between Grades" for more information on dry cleaning, or rather on purging the sampling system with coal.

7.4. Upon completion of the loading of a vessel and after removal of the assay and moisture samples, the sampling system should be inspected with feeders and belts running. This inspection ensures that wet coal is removed from the system before it freezes solidly and plugs chutes or cutters.

7.5. During freezing weather one (1) of the two (2) hydraulic systems should be run in the manual mode.

- cont'd -

8.0. GENERAL REMARKS

8.1. During loading of a vessel washing and cleaning with water inside or near the sampling plant is not permitted to avoid contamination of moisture samples.

8.2. Plan washing and cleaning carefully so that the first lot of a consignment that is loaded aboard the next vessel, is not biased due to the excessive amounts of water in secondary system for moisture samples. At least four (4) hours must elapse between completion of cleaning and commencement of loading to ensure that the sampling system is dry.

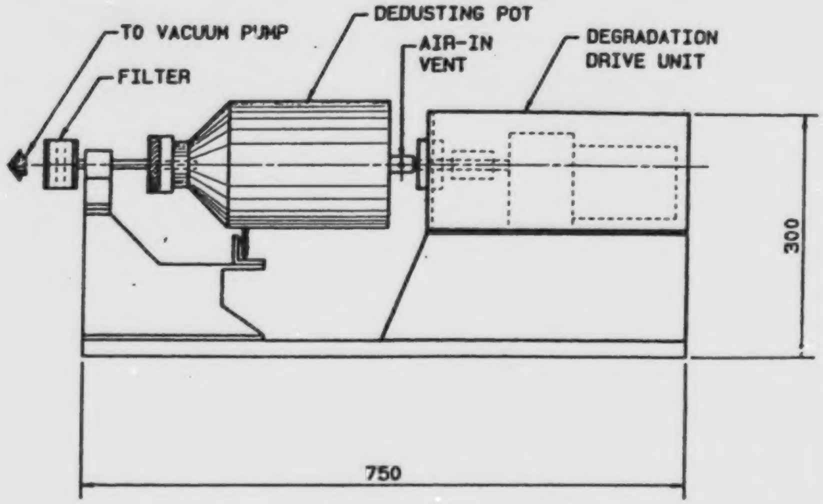
8.3. Check the sampling system at regular intervals and be alert for plug-ups in hoppers and feeders, in particular during a secondary cycle. Remove gross samples for moisture and for assay from the hoppers as soon as the lot and the last secondary cycle are finished.

8.4. Remove and store gross samples if a switch to another consignment of coal is made. If a lot is nearly completed (less than 1,000 mt to go), the samples for this partial lot should be processed, and the difference in tonnage added to the next lot.

8.5. Always purge the sampling system between different grades.

8.6. Components of the sampling systems may fail at random. Report failures that cannot be corrected, as soon as possible. Describe which part of the system broke down, and which component failed. Make notes on the status report for the system and on the data forms for the consignment. Inform the foreman during the night shift, the maintenance supervisor Mr. Gary Wilton during the day, and the supervisor of the inspection company, to ensure that the sampling system will be repaired as soon as possible.

8.7. Complete and sign data forms for all lots so that a loading log for each consignment can be compiled. All data are important!!


TO VACUUM PUMP
FILTER
DEDUSTING POT
AIR-IN VENT
DEGRADATION DRIVE UNIT
300
750

GENERAL SPECIFICATIONS

-DEDUSTING POT -152 MM. INS. DIAMETER
-MAIN BODY 200 MM. LONG, CONED @ 45 DEG.
C/W (3) - 12 MM. HEIGHT FLIGHTING
-50 MM. PIPE OPENING C/W SCREW CAP
-OVERALL LENGTH 300 MM.
-WEIGHT 3.3 KG.
-STAINLESS STEEL CONSTRUCTION

-POT REVOLUTION @ 33 RPM.

-FILTER - .45 um

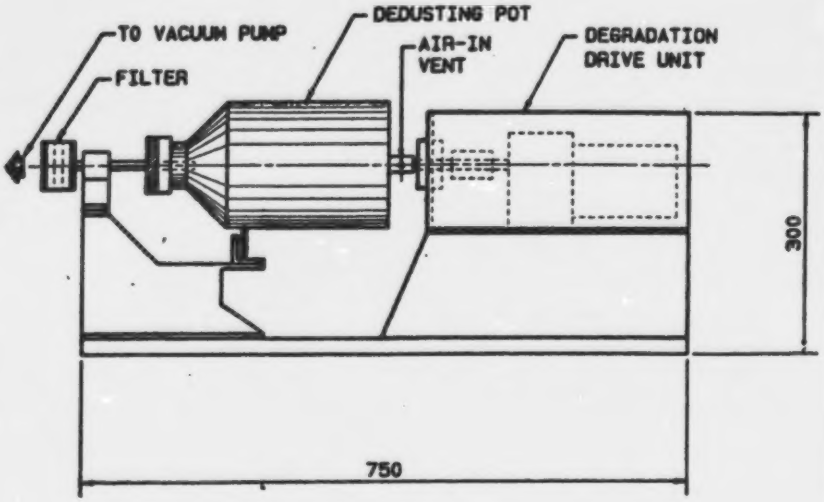
-VACUUM PUMP - INITIAL PRESSURE 15 psi.

PCS
Mining

Figure 2. Design of the PCS Dedusting Test Equipment

APPENDIX XV

PCS DEDUSTING APPARATUS


TO VACUUM PUMP
FILTER
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-POT REVOLUTION @ 33 RPM.

-FILTER - .45 um

-VACUUM PUMP - INITIAL PRESSURE 15 psi.

PCS
Mining

Figure 2. Design of the PCS Dedusting Test Equipment

DEDUSTING PROCEDURE

When applying this procedure to GRANULAR and COARSE product, first screen to -6+10 mesh. For STANDARD product - proceed as follows:

1. Transfer 200 grams of product to the dedusting pot along with fifty (50) 5/16" steel balls.
2. Weigh the filter apparatus.
3. Attach the filter to the vacuum pump and adjust the vacuum to 15 psi.
4. Rotate for 15 minutes with vacuum pump running.
5. Weigh the filter apparatus and calculate ppm dust.
6. Wash the pot with methanol after tests with reagentized products.

APPENDIX XVI

IMCC RESEARCH REPORT
RELATIVE HUMIDITY VERSUS % MOISTURE ON PRODUCTS

INTERNATIONAL MINERALS & CHEMICAL CORPORATION (CANADA) LIMITED

RELATIVE HUMIDITY VERSUS % MOISTURE ON PRODUCTS

A humidity chamber was set up to try to determine the levels of moisture our products would reach under different relative humidity conditions.

There were problems in controlling the temperature and relative humidity due to poor control on the humidistat and wide fluctuations in the room relative humidity and temperature. However, the trends are indicative of what can be expected in product storage.

Results were plotted to eliminate any stray data for each product and a table made up.

The Ag. White used was anticaked and significantly absorbs less moisture at any relative humidity. Special Standard is the most vulnerable product, levelling off at 0.45% moisture at a low 20% relative humidity. All products when maintained at 90% relative humidity became soaked in water.

Presently dryer temperatures are maintained at 143-225°C resulting in moistures of less than 0.05%. Within 4 hours of cooling the Coarse will be at 0.10% moisture, the Standard at 0.25% and the Special Standard at 0.41% with a relative humidity condition of 20%. This seems uneconomical with the only apparent purpose to flash off any moisture on the existing storage pile.

Unanswered questions are, to what depth in a pile will moisture be absorbed and to what extent does treating material prior to storage change the situation.

Victor Brace
Plant Metallurgist
February 17, 1988

Esterhazy, Saskatchewan S0A 0X0 Telephone 745-3931

DEDUSTING PROCEDURE

When applying this procedure to GRANULAR and COARSE product, first screen to -6+10 mesh. For STANDARD product - proceed as follows:

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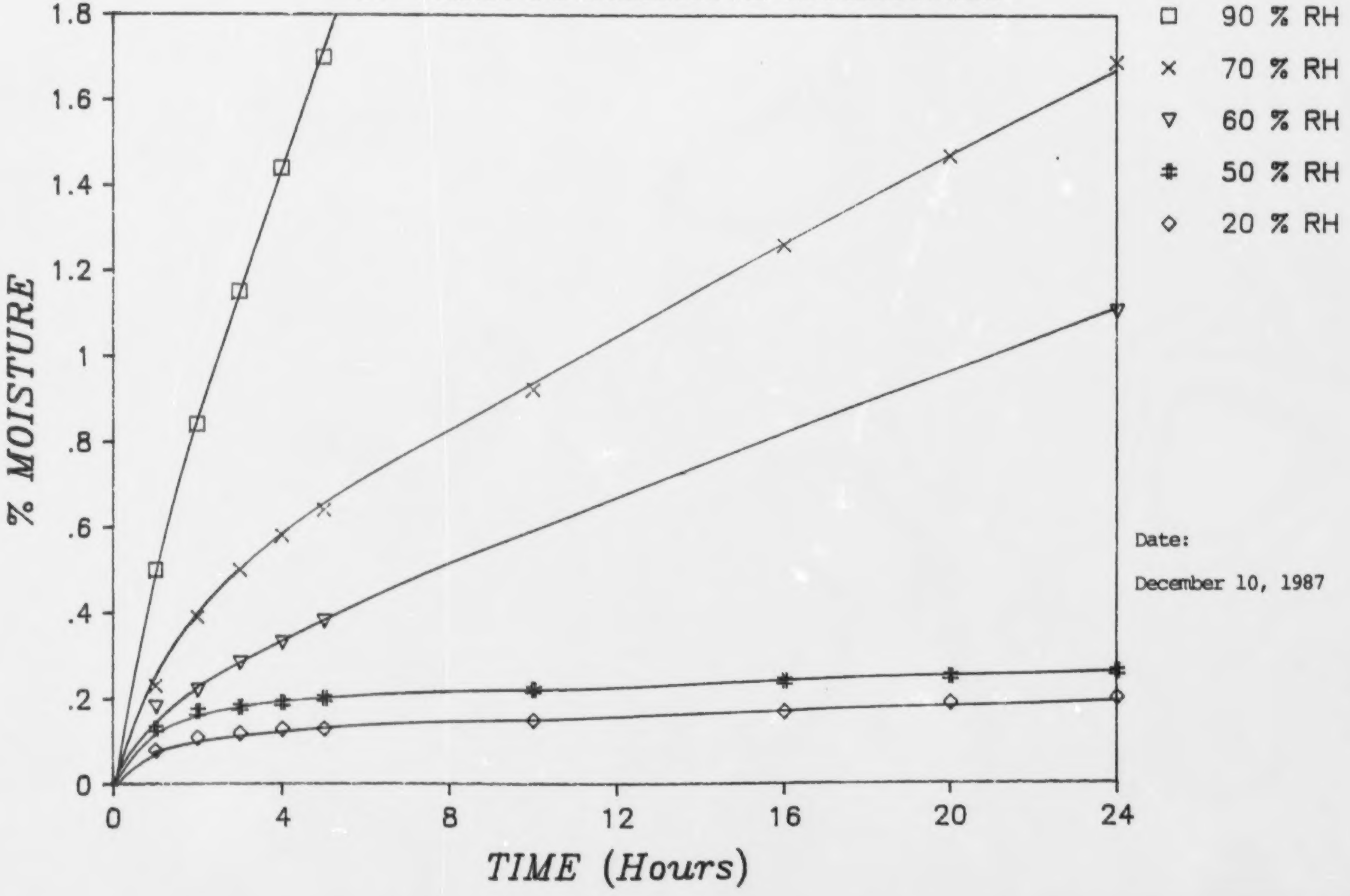
K−1 GRANULAR

at VARIOUS RELATIVE HUMIDITIES

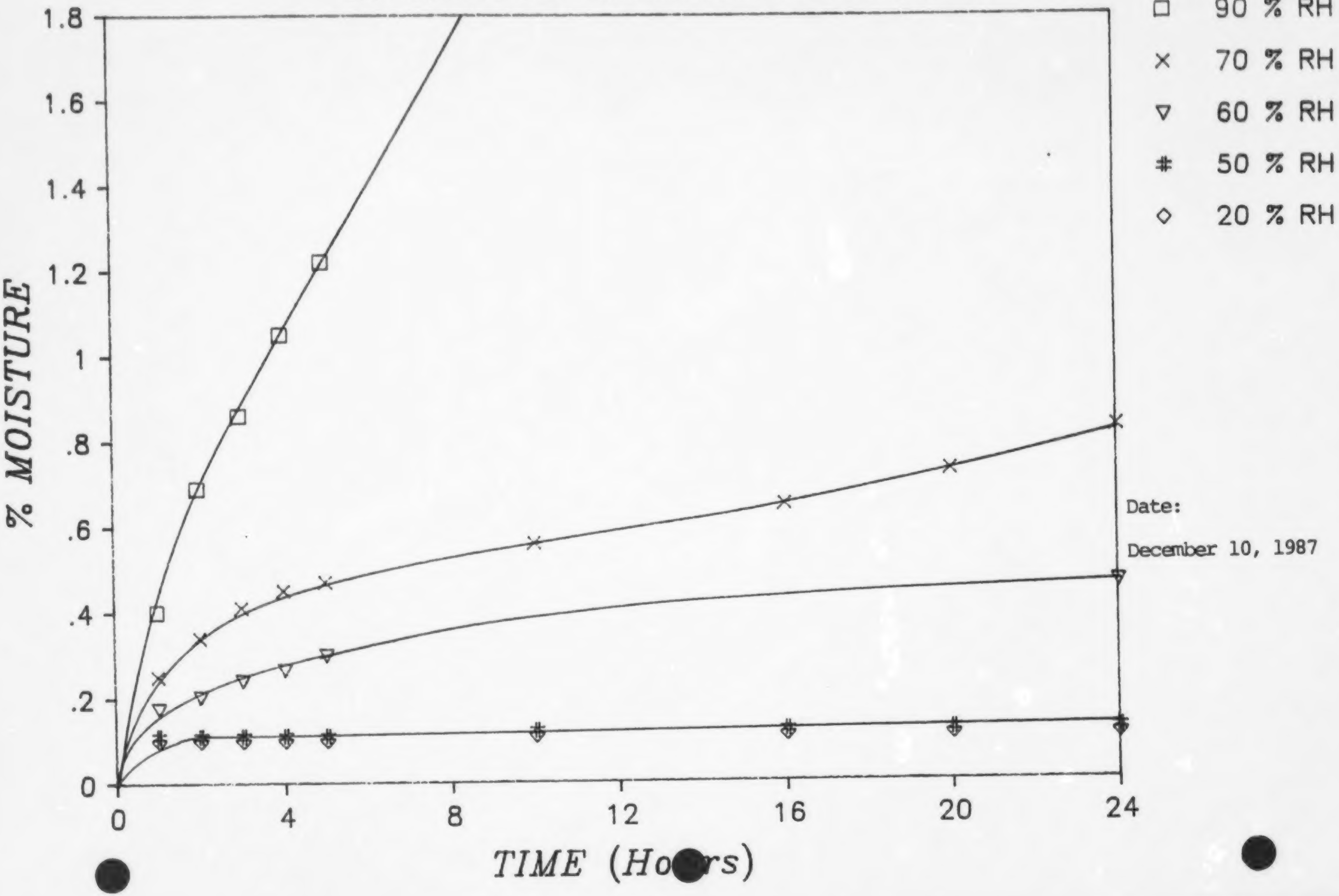


Date:

December 10, 1987

K–1 COARSE

at VARIOUS RELATIVE HUMIDITIES

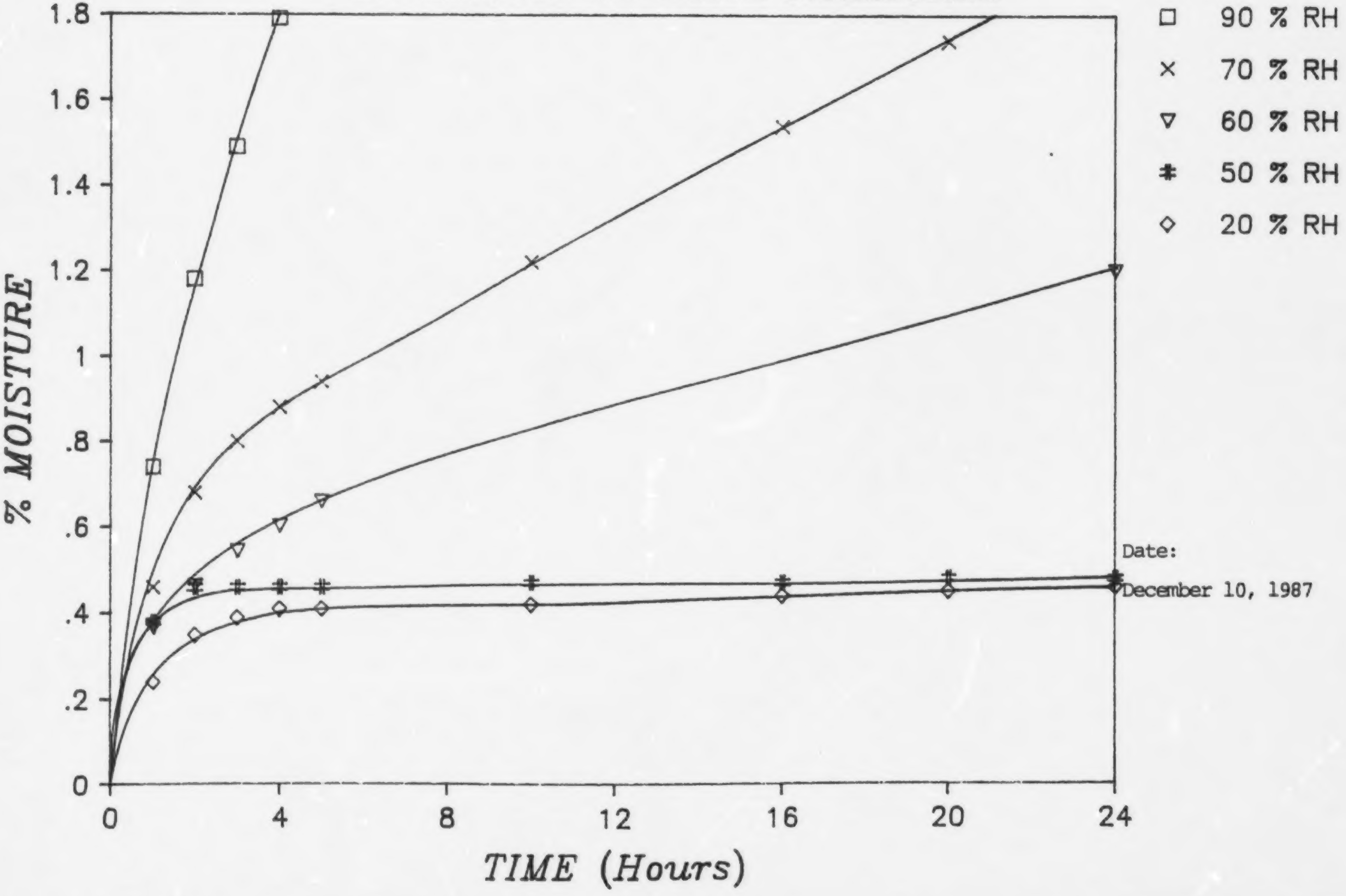


Date:

December 10, 1987

K−1 SPECIAL STANDARD

at VARIOUS RELATIVE HUMIDITIES



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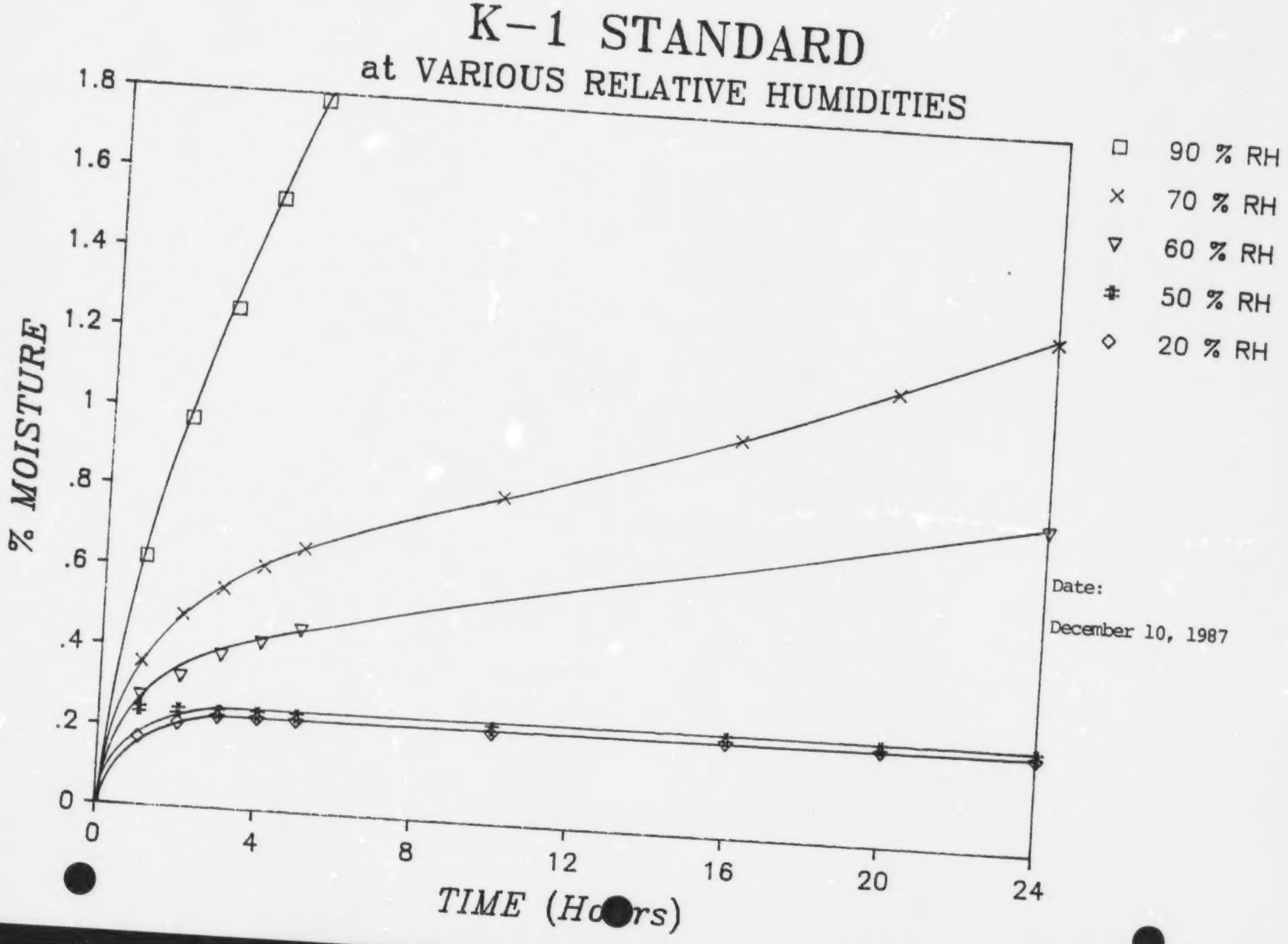
December 10, 1987

# K−1 STANDARD

at VARIOUS RELATIVE HUMIDITIES

Date:
December 10, 1987

APPENDIX XVII

IMCC RESEARCH REPORT
TREATMENT EFFECTS ON PRODUCT MOISTURE

INTERNATIONAL MINERALS & CHEMICAL CORPORATION (CANADA) LIMITED

TREATMENT EFFECTS ON PRODUCT MOISTURES

Reference: February 17, 1988 report "Relative Humidity Versus % Moisture On Product"

Further testing was done to determine the effects of treating our products to prevent moisture pick-up with time at high humidity conditions.

These products were treated with a 3% Armeen T in #1000 oil mixture and compared with untreated products. A relative humidity of 70% was used.

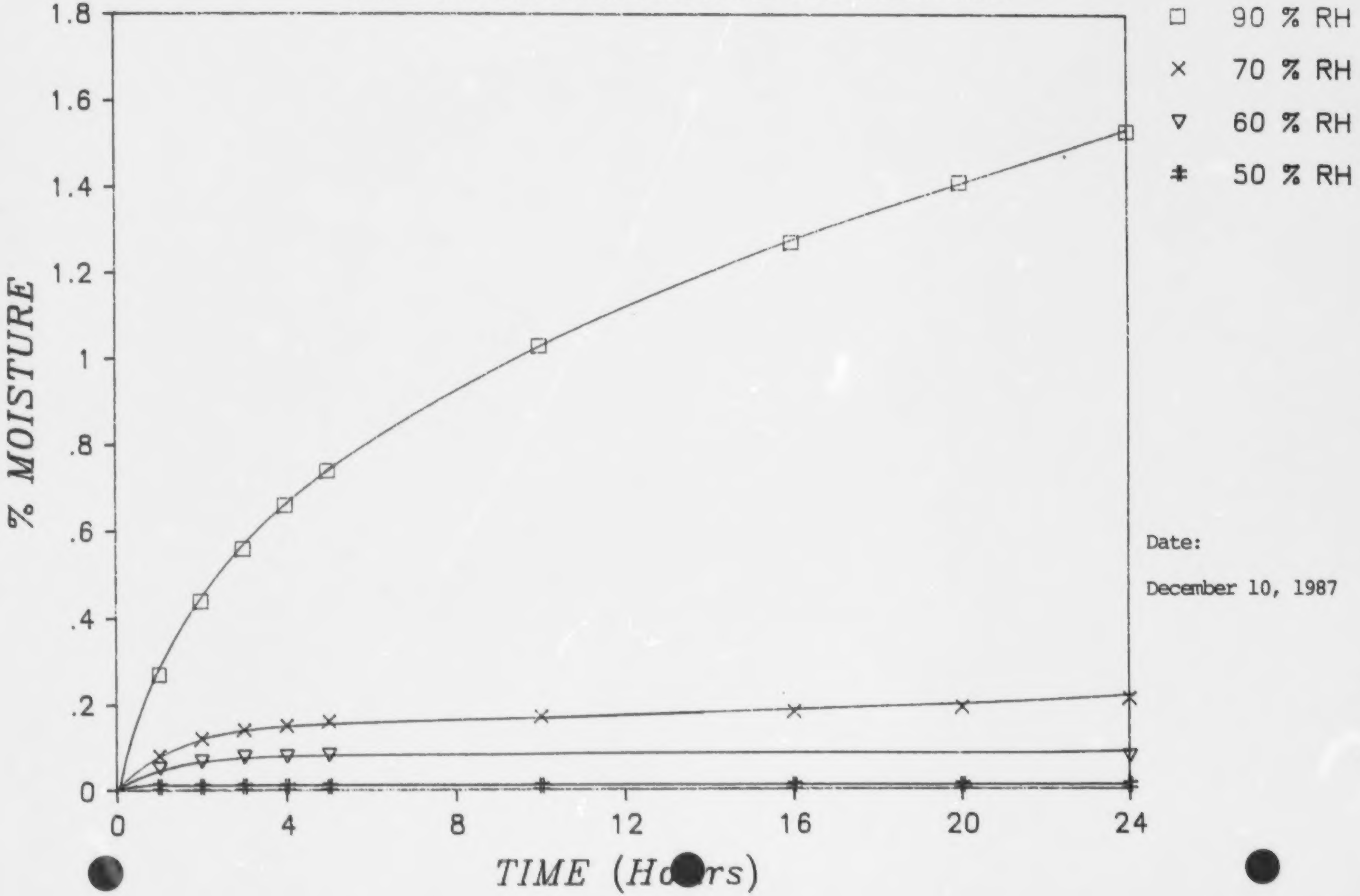
Treating the products did not reduce the amount of moisture they picked up while at high humidity. Except for Granular, the treatment actually worsened the condition. On Granular, the % moisture remained practically the same for both.

Victor Brace
Plant Metallurgist
February 29, 1988

Esterhazy, Saskatchewan S0A 0X0 Telephone 745-3931

K–1 AG. WHITE ANTI-CAKED

at VARIOUS RELATIVE HUMIDITIES



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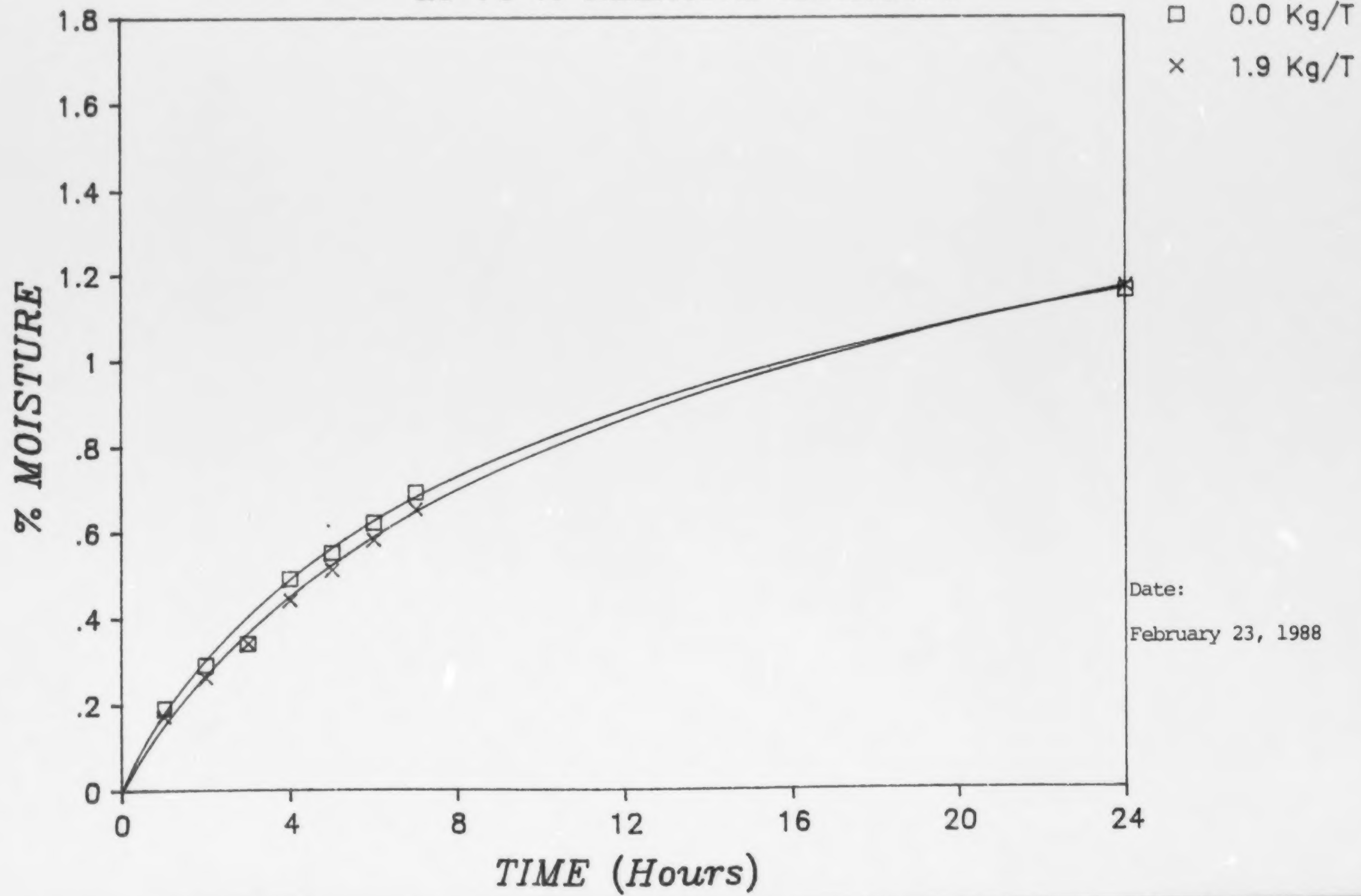
December 10, 1987

K–1 GRANULAR
at 70 % RELATIVE HUMIDITY

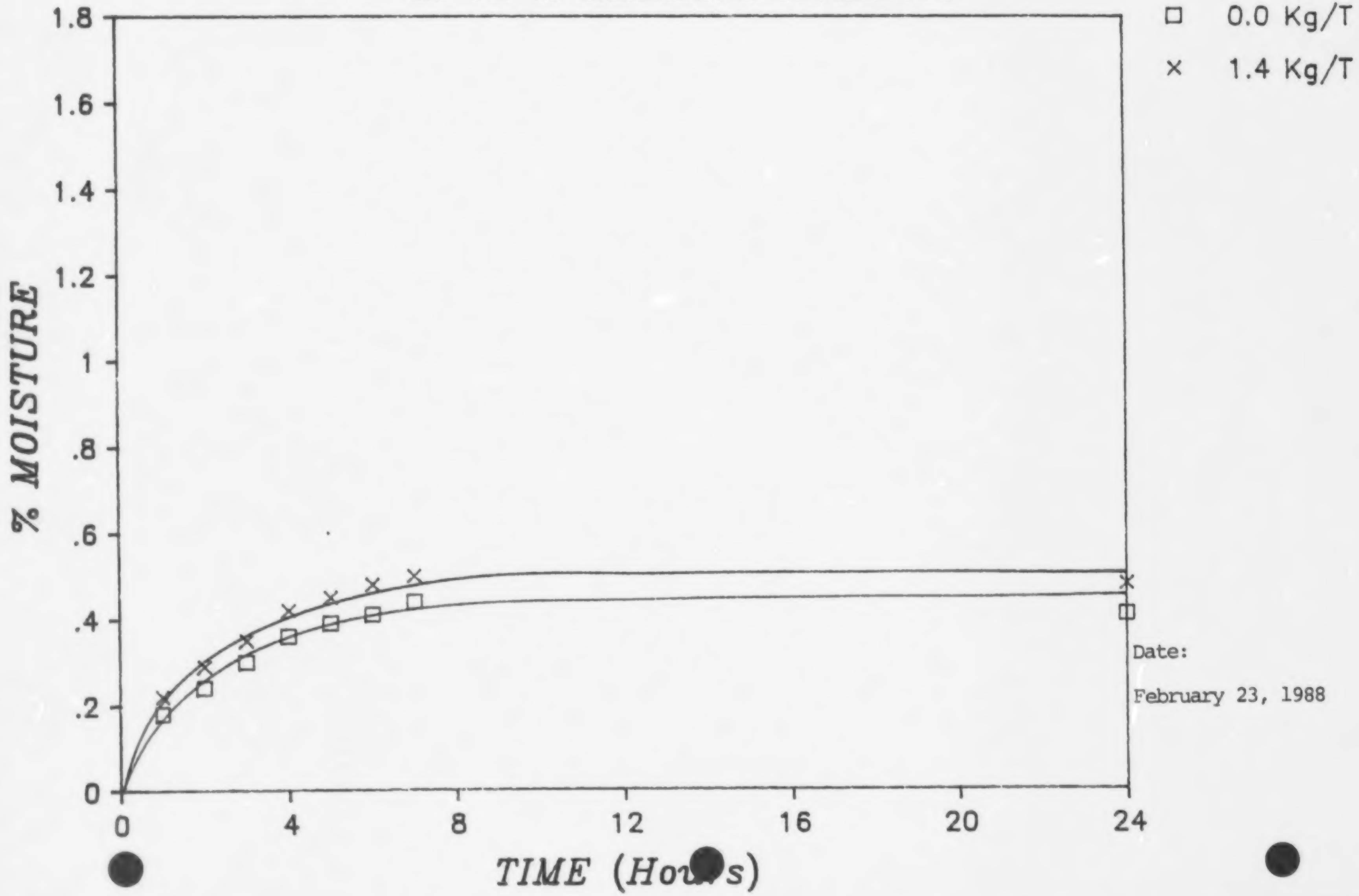


Date:

February 23, 1988

K–1 COARSE

at 70 % RELATIVE HUMIDITY

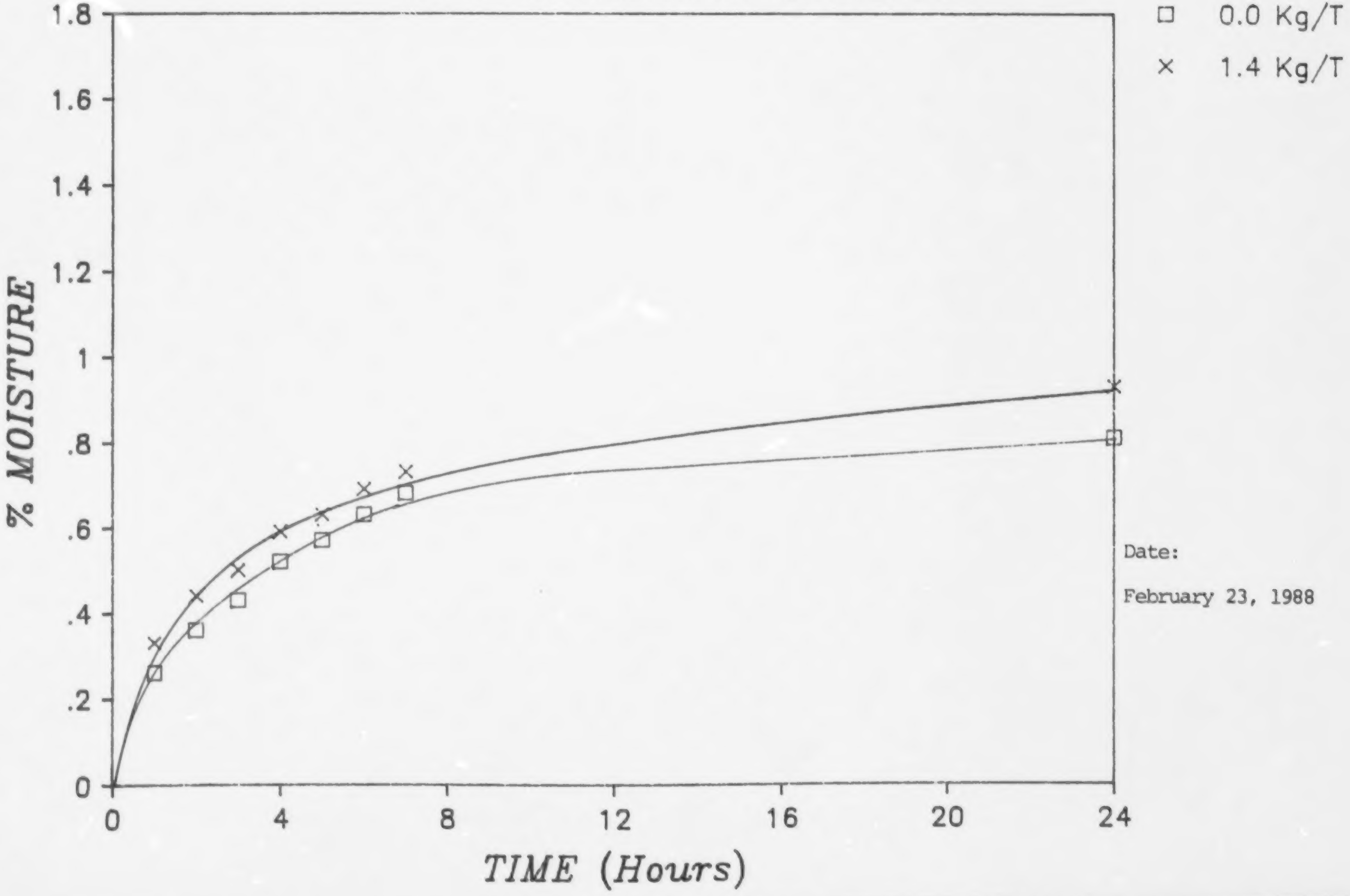


Date:

February 23, 1988

K−1 STANDARD

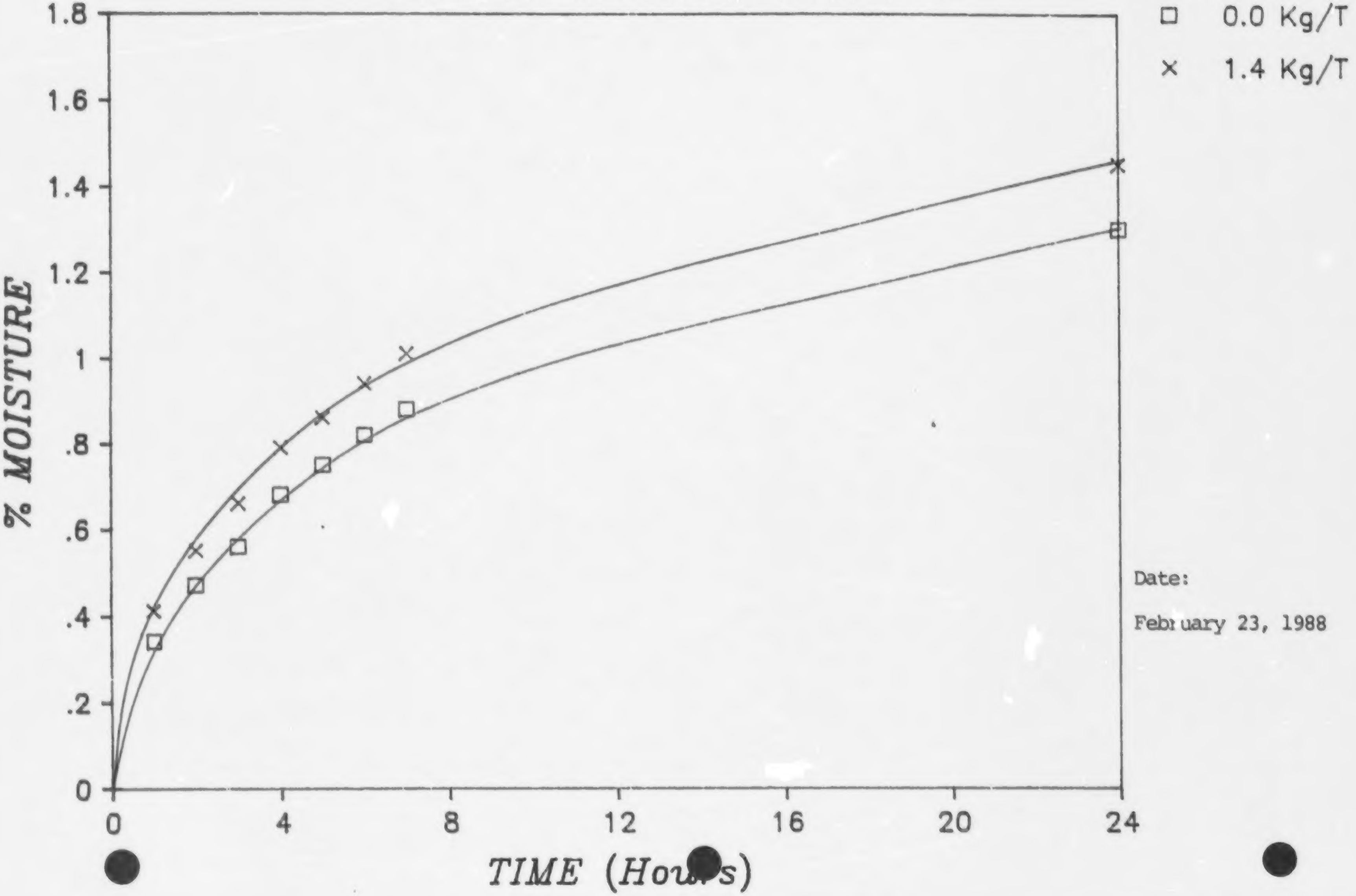
at 70 % RELATIVE HUMIDITY



Date:

February 23, 1988

K-1 SPECIAL STANDARD
at 70 % RELATIVE HUMIDITY



Date:

February 23, 1988

APPENDIX XVIII

DEVICE MEASURES WHEAT HARDNESS

Device Measures Wheat Hardness

When ground, one by one, wheat grains give off vibrations that scientists can measure to test for hardness, a key to grading and pricing.

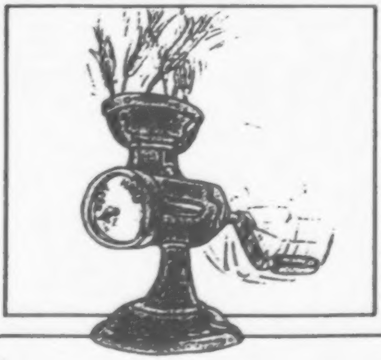
The measuring device, invented by engineers David R. Massie and Karl H. Norris of USDA's Agricultural Research Service, electronically registers grain vibrations produced by grinding. A computer linked to the device prints hardness scores on a readout tape.

"Hard grains grind slightly louder than soft ones," says Norris. "The simplicity of the device could make it especially valuable to grain inspectors. If adopted nationally, it could eventually help raise both the quality of domestic wheat flour and the value of our wheat exports."

Hard wheats bring a higher price for flour, while soft wheats generally sell for less. "Wheat breeders have told us that a quick measure of wheat hardness will help them evaluate new varieties," Norris explains. "This would help seed companies, too, and be even better for farmers who will know exactly what they are planting and marketing."

A sample of grain can be tested by pouring it into a feeder trough that sets on top of a 6-inch diameter cylinder. The electrically operated device vibrates the grains in the trough so they fall one at a time from the edge of the trough into the cylinder. There, a high-speed rotor hurls each grain against an outer ring with a rough surface. The impact crushes each grain.

USDA's Federal Grain Inspection Service is testing the latest grinder model for possible use in wheat grading. The agency sets guidelines for agricultural commodity graders and issues licenses to grain inspectors. ☐



APPENDIX XIX

MINOLTA CHROMAMETER CR-200

MINOLTA CHROMA METERS
For measuring reflected- or transmitted-light color

Minolta tristimulus colorimeters provide color and color-difference measurements from a wide variety of samples. Compact and lightweight, these durable instruments are ideally suited for high-volume use in both production and laboratory applications.

Color and color-difference readings

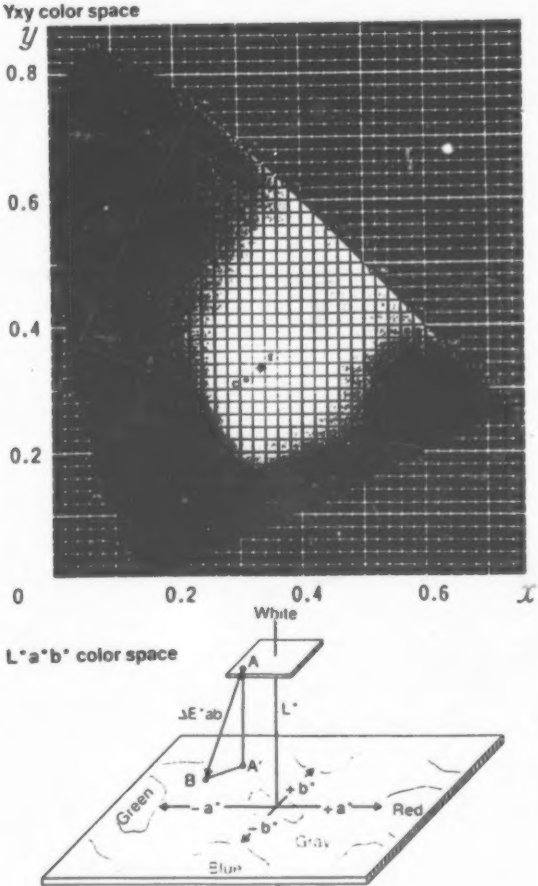
Minolta Chroma Meters provide highly accurate measurements of chromaticity and illuminance, and also enable quick, objective comparisons between two samples for matching colors or setting tolerances.

CIE standard illuminant (Illuminant C or D_{65} selectable) and observer conditions are used when measuring the sample's spectral characteristics, enabling determination of X, Y, and Z tristimulus values.

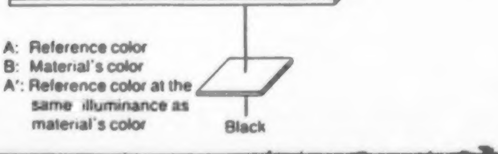
For color readings, these values are translated into Yxy coordinates (accepted internationally as the standard in color communication) or $L^*a^*b^*$ coordinates, whose spacing correlates more closely with color change as perceived by the human eye.

For color-difference readings, data is displayed in terms of Δ (Yxy), Δ ($L^*a^*b^*$), or ΔE^*ab. Readings can be converted instantly from one color system to the other at the push of a button.

Yxy color space



L*a*b* color space

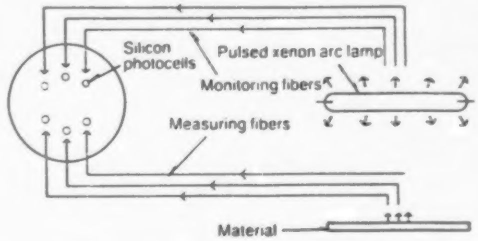


A: Reference color
B: Material's color
A': Reference color at the same illuminance as material's color

Consistent, diffuse illumination

Minolta Chroma Meters use a pulsed xenon arc (PXA) lamp inside a mixing chamber to provide diffuse, even lighting over the sample. A double-beam feedback system is also incorporated to ensure that lighting remains consistent for each measurement.

In addition to the sensors which measure the light from the sample, three other photocells read the color of the incident light each time the lamp is fired. The microcomputer then uses this data to check and compensate for any changes in the illumination.



Measuring system

The measuring system employed by these colorimeters is designed to provide accurate readings and uniform response. Light from the PXA lamp evenly illuminates the sample, and only that reflected (or transmitted) along the normal to the surface is collected by the optical cable for color analysis.

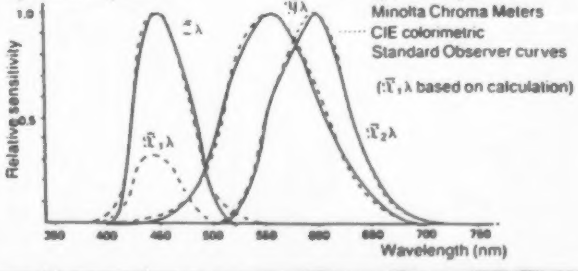
The light received is divided three ways and passed through special filters whose light-absorbing characteristics combine with the spectral response of the photocells to duplicate CIE 1931 Standard Observer functions.

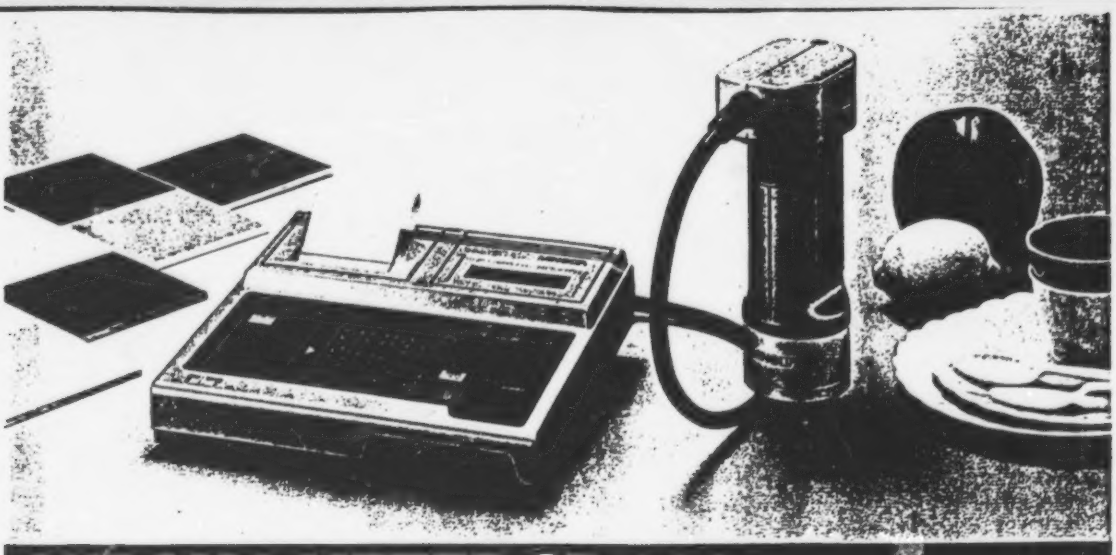
Upon striking the silicon photocells, light energy is converted into electrical signals and sent to the microprocessor, where it is adjusted for the illuminating condition desired and then converted into coordinates for the chosen color space.

Readings are displayed in the LCD panel and can be transferred to a separate computer or processor through the data-output terminal.

- Minolta also sells incident-light Chroma Meters for measuring the chromaticity and color temperature of light sources and other luminous objects.

Spectral response





CHROMA METER CR-200

The general-purpose colorimeter for spot measurement, with a built-in data processor and an 8mm-diameter measuring area, it uses diffuse illumination, 0° viewing angle geometry to provide accurate readings in a wide variety of color-control applications.

Main features

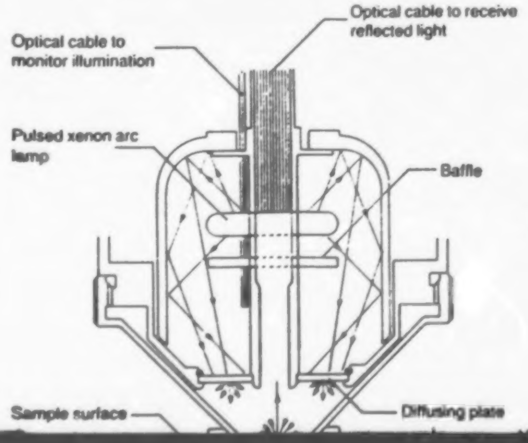
- **Precise readings with high repeatability**
  The latest advances in microprocessing are integrated with time-tested technology developed over years of colorimeter manufacturing.
- **Built-in data processor and thermal-dot printer for greater versatility.**
  Measurements are displayed on an LCD panel. A thermal-dot printer is also built in to enable data printout.
- **Readings displayed in four color-space notations plus colorimetric reflection density**
  Chromaticity and color difference readings displayed in Yxy, L*a*b*, L*C*H°. Chromaticity reading displayed in Munsell. Colorimetric reflection density can also be measured.
- **Choice of calibration standards**
  Calibration can be done using the white plate supplied or other reference selected by the user. This enables a number of meters to be standardized for consistent response.
- **Data storage with memory backup**
  Built-in memory provides space for 20 calibration standards, 20 target colors for color-difference measurements, and 300 sets of measured color values.
- **Statistical calculations**
  Built-in data processor performs statistical calculations, providing maximum, minimum, mean, and standard-deviation values of data in memory.
- **Automatic measurement at user-selected intervals**
  The built-in timer can be set for automatically taking measurements at selected intervals.
- **Alarm function**
  The alarm can be used to warn users when color difference is outside preset limits.
- **Choice of two illuminants**
  Either CIE Illuminant C or D_{65} can be selected.
- **Data-output terminal**
  RS-232C format output terminal is provided for connection to another computer.

Applications

- **Manufacturing:**
  Color control of painted and coated surfaces
  Color control of rubber and plastic parts
- **Printing:**
  Control of color density and ink characteristics
- **Textiles:**
  Color monitoring of thread, fabric, etc.
- **Agriculture:**
  Color analysis of soil, fruits, grain, etc.
- **Molding:**
  Color control of ceramic and glass products
- **Medicine:**
  Color analysis of skin, tissues, etc.

Illuminating system

The CR-200 uses diffuse illumination, 0° viewing angle geometry to obtain a reading that correlates well with color as seen under typical lighting conditions. Since illumination both in- and outdoors is usually diffused, this system employs a mixing box and diffusing plate to project light evenly onto the sample from all angles. Light reflected vertically from the 8mm-diameter measuring area is picked up by the optical cable for color analysis.


Optical cable to receive reflected light
Optical cable to monitor illumination
Pulsed xenon arc lamp
Baffle
Sample surface
Diffusing plate

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- Totally Controlled Environment Monitoring 5
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- Microscopic Analysis 17
- Interpretation and Conclusion 27

APPENDICES

I Climate Chamber Installation and Product Handling

II Conductivity Probe Evaluation

III Investigation of Moisture Measurement in Processed Potash Using Microwave Techniques

IV Monitoring Results and Graphs - First Monitoring Period

V Monitoring Results and Graphs - Second Monitoring Period

INTRODUCTION

The objectives of this study can be described as follows:

1. Develop a more scientific approach (as opposed to empirical) in studying potash product behaviour, when exposed to extreme temperature and humidity conditions.

2. Measure and analyze the actual changes in potash material that occur between mine sites and customers, including effect of storage on material properties.

3. Increase knowledge of fundamental materials science of potash product by identifying heat and moisture migration patterns and transport mechanisms within potash piles. This will provide a better understanding of the product caking process, and identify "shelf-life" factors influencing product quality.

To achieve these goals, it was proposed (Study Proposal Heat and Moisture Transport in a Potash Pile, dated November 20, 1987) to monitor the variables believed responsible for potash product caking in a totally controlled environment and in the field. The variables to be monitored were identified as follows:

- Ambient temperature
- Product temperature
- Ambient relative humidity
- Relative humidity inside the pile (void space)
- Product moisture
- Internal pressure
- Time

Initially, it was intended to execute this project in four different phases, listed as follows:

Phase I : Instrumentation evaluation
Phase II : Totally controlled environment monitoring
Phase III: Mine site/railcar monitoring
Phase IV : Pile monitoring at Canpotex storage facilities

Phases I and II are presently completed, and the collected data, interpretation of results and recommendations for future work are presented in this report. Phases III and IV have been postponed until the present results are reviewed.

INSTRUMENTATION EVALUATION

This phase consisted of testing and evaluating the instrumentation and equipment required to monitor the variables responsible for potash product caking as described previously, in both a controlled and an uncontrolled environment.

Ambient Temperature and Relative Humidity

The measurement of the ambient temperature and ambient relative humidity did not present any specific problem. Both variables could be measured very easily using simple and already available instrumentation. In the climate chamber (totally controlled environment) dry and wet bulb Resistance Thermometer Detectors (RTD) were used to monitor continuously the ambient temperature and ambient relative humidity. For rapid reference and control a Cole Parmer 4-speed Hygrothermograph, Model 8368-60, was also used. This Hygrothermograph records temperature from -15 to +50°C and relative humidity from 0 to 100% with accuracies of ± 1°C and ± 3% respectively. The variables can be registered on a single chart for periods of 1, 7, 31 and 62 days.

Product Temperature and Relative Humidity

The measurement of product temperature and relative humidity inside the pile (in the void space) necessitated a more careful review and evaluation of the instrumentation currently available on the market, due to the specific testing and environmental conditions, and the physical and chemical properties of the potassium chloride products.

The product lines of several instrumentation manufacturers were carefully and thoroughly reviewed. The Vaisala HMP 135Y humidity and temperature probes and transmitter met all our requirements and was chosen for its versatility and accuracy. The probe measurement range is 0 to 160°C and 0 to 100% relative humidity, with an accuracy of ± 0.2°C at +20°C and ± 2% RH between 0-90% RH, and ± 3% RH between 90-100% RH. The probe itself is enclosed in a brass sintered filter with openings of 133 microns diameter. The transmitter is protected by a Nema 4 type of enclosure, therefore it could be buried safely under potash material. (See pictures in Appendix I.) Hoskin Scientific Ltd. of Vancouver is the Canadian distributor of Vaisala equipment.

Product Moisture

The in situ measurement of the product moisture at different locations inside the potash pile presented the biggest challenge. The instrumentation technology to measure accurately the moisture level in the 0.03% to 0.40% magnitude is not yet available on the market. Therefore, to overcome the problem, the possibility of correlating the moisture level with other variables, such as product conductivity, dielectric constant and microwave resonant effects has been investigated.

A conductivity probe (Wenner arrangement) borrowed from the University of Saskatchewan was used to test the correlation between product conductivity (resistivity) and the moisture level. Unfortunately, the attempt was unsuccessful. Too many other uncontrolled and uncontrollable variables, such as product bulk density, product size distribution, etc. affected the conductivity (resistivity) level of the product. The testing and evaluation procedures are presented in Appendix II.

The Centre for Communication Studies, Department of Electrical Engineering, University of Saskatchewan, was contracted to study the correlation of the moisture level with the product dielectric constants and the resonant effects. Dr. Kumar identified a positive correlation between the surface moisture level (in percent) and the dielectric loss in each potash material. However, too many other variables are somewhat related to the dielectric loss, such as occluded moisture, bulk density, particle size distribution, treatment reagent type and dosage. A multivariate model integrating all these variables is not available at this time. Therefore, these microwave techniques cannot be used for this study. A copy of Dr. Kumar's report and my comments and recommendations for future development are presented in Appendix III.

Instrumentation or technique for in situ measurement of product moisture has not been identified. Therefore, for the Totally Controlled Monitoring Phase, product samples were taken manually on a daily basis using a 3-foot long probe, and moisture was analyzed by gravimetric method.

Internal Pressure

For product monitoring in railcars and storage locations, it was proposed to estimate the internal pressure using the formula:

$p = hd$

where p = internal pressure in the product
h = height of product above the reference point
d = bulk density of the product.

The pressure estimation was judged adequate considering that the pressure is applied in hydraulic fashion and that the bulk density does not vary significantly across the entire pile.

TOTALLY CONTROLLED ENVIRONMENT MONITORING

This phase consisted of measuring all the known variables for product caking in a pile of well defined geometry and in a rigorously controlled temperature and relative humidity environment. To meet these requirements a climate chamber and permeable containers have been built in the PCS Pilot Plant.

Climate Chamber

The climate chamber consists of a modified garden shed with the following characteristics (see Figure 1 and pictures in Appendix I).

Length: 3.95 metres (13.0 feet)
Width : 2.90 metres (9.5 feet)
Height: 2.20 metres (7.2 feet)

All four walls, the floor, ceiling and doors are insulated and sealed as much as possible with blue 2.54 cm (1 inch) insulating styrofoam and duct tape.

The chamber is maintained under a positive pressure of 0.13-0.15 cm of water (0.05-0.06 inches). To uphold this pressure, a net air inflow of about 47 litres per second (100 cfm) is required. This 47 litres per second of air is fresh air from outside the Pilot Plant. To ensure an adequate distribution of air in the chamber an additional 141 litres per second (300 cfm) of air is recirculated.

The effective temperature that can be maintained in the chamber is the pilot plant outside air temperature and 40°C with an accuracy of ± 1°C. In Winter, a sub-freezing temperature can be attained, but is not recommended due to equipment freezing problems. The use of outside air is to eliminate the need for a refrigeration unit and to maintain costs at a minimum level. The heat is provided by a home-made electric heater that was designed and used for another project, but that is largely overrated for the present experiments. The temperature is controlled by a "Penn" temperature controller (0 to 100°F at ± 2°F).

From the outside air relative humidity to about 60%, the moisture is supplied by a portable apartment humidifier, Electrohome EHR30, in which the water temperature was maintained at a relatively constant 30°C. With the control provided with the unit, the relative humidity level can be maintained with an accuracy of about ± 2% RH. From 60% to 100% RH, the additional moisture is provided by low temperature (105-110°C) steam and is controlled by a "Penn" relative humidity controller (10-100% RH ± 5%). (See pictures in Appendix I.)

All the monitored variables in the product (temperature and relative humidity) and the climatic conditions of the chamber are recorded, using a data logger Acro 900 system from Acrosystems and a Soltech personal computer.

To monitor the relative humidity and temperature transport in piles of well defined geometry, two cylindrical permeable containers are used. The containers have a 4-foot diameter and are four feet high. To let the moisture and heat transfer freely from the atmosphere to the product, and vice versa, the containers were built with 20 mesh stainless steel screen cloth material. Each container can hold about 1.5 to 1.7 tonnes of potash depending on the bulk density of the product tested (see picture in Appendix I).

Monitoring Periods

The monitoring in a totally controlled environment (in the climate chamber) was done over two different periods, each one with a different temperature setting. During each period the temperature was maintained constant and relative humidity was changed periodically. The following is a description of the objectives and environmental conditions for these two monitoring periods.

A. First Monitoring Period

The objectives of this first monitoring period were twofold. The first was to confirm that the Vaisala HMP 135Y temperature and relative humidity probe and transmitter were adequate to monitor the intended variables over a long period of time, and relatively harsh conditions. The second objective was to start gathering data on moisture and heat transfer in potassium chloride products.

The first monitoring period lasted 42 days and the conditions were set as follows (in chronological order):

Temperature maintained constant at 15 ± 2°C

- 9 days : RH at 52 ± 3%
- 9 days : RH at 72 ± 3%
- 12 days: RH at 87 ± 3%
- 12 days: RH at 52 ± 3%

The product used for this experiment was PCS Mining, Cory Division Granular 210, treated with dedusting and anticaking reagents at the same level as normal Cory shipment.

1.2 kg/tonne Husky Oil S-288
0.2 kg/tonne Armeen "T"
0.04 kg/tonne Glycol

B. Second Monitoring Period

The second monitoring period lasted a total of 66 days, and its objectives were:

1. Repeat some experiments of the first monitoring phase to confirm reproducability of the data.

2. Observe and analyze behaviour differences between products of different size and composition.

   Granular (-4 +20 mesh) mainly compacted material
   Standard (-10+65 mesh) mainly natural crystal

3. Measure and analyze changes that occur in potash material that has been exposed to several cycles of low-high relative humidity in a high temperature environment.

For this 66 day monitoring period, the conditions were set as follows:

Temperature maintained constant at 27 ± 2°C

- 14 days: RH at 57 ± 3%
- 14 days: RH at 75 ± 3%
- 6 days : RH at 87 ± 3%
- 11 days: RH at 52 ± 3%
- 7 days : RH at 85 ± 3%
- 14 days: RH at 52 ± 3%

Cory Granular 210 and Standard products were used in this experiment. Both products were treated with dedusting and anticaking reagents at the same level as normal Cory shipment.

Granular 210: 1.2 kg/tonne Husky Oil S-288
0.2 kg/tonne Armeen "T"
0.04 kg/tonne Glycol
Standard : 1.0 kg/tonne Husky Oil S-288
0.2 kg/tonne Armeen "T"
0.07 kg/tonne Glycol

For both monitoring periods, treated products were chosen to reflect as accurately as possible product changes and behaviours in normal shipment conditions.

C. Instrumentation and Sampling

For both experiments, two Vaisala HMP 135Y probes monitoring relative humidity and temperature simultaneously were installed in each permeable container (product). One probe was buried 24 inches deep into the pile (measured from the top of the container) and 6 inches in (measured from the side wall of the container). The second one was located at the centre, 24 inches deep and 24 inches from the wall.

For moisture (water absorption) monitoring, product samples were taken in the vicinity of each probe and at the surface of each pile, on a daily basis. The moisture level of each sample was determined by gravimetric method analysis.

MONITORING RESULTS

I FIRST MONITORING PERIOD

The first monitoring period lasted 42 days, during which the temperature was maintained constant at 15 ± 2°C. The relative humidity was changed periodically from 52 to 87%.

Note: All the data and the graphs showing the rate of change of all the monitored variables are shown in Appendix IV.

52% Relative Humidity

During the first 9 days, the relative humidity was maintained at 52 ± 3%. Originally the product was in equilibrium with the pilot plant climatic conditions, which can be listed as:

Temperature : 22.5°C
Relative humidity: 12%
Product moisture : 0.037%

During this period, the temperature of the product and the relative humidity of the air trapped in the pile departed from the pilot plant conditions to reach 15°C and 40-42% RH. The product moisture of the entire pile also increased to 0.075%. The temperature and the relative humidity changes seem to follow an exponential function, while the product moisture changes appear to be linear.

72% Relative Humidity

During the following 9 days, the climate chamber relative humidity was maintained at 72 ± 3%. After this period the product seemed to reach a new equilibrium: temperature 15°C, relative humidity 45-48% and moisture of 0.10-0.12% through the entire pile.

87% Relative Humidity

During the next eleven days the relative humidity in the climate chamber was maintained at 87 ± 3%, which is significantly higher than the critical relative humidity of the potassium chloride (83%). Under these conditions the relative humidity inside the pile increased very slowly to level out at about 62-65%. The product moisture at the surface of the pile increased very rapidly to a 4.2-4.8% level, while the moisture inside the pile (at least 6 inches deep) increased very slowly but constantly to 0.17-0.23% range. As could be expected in conditions above the critical relative humidity, the moisture absorption of the product did not reach an equilibrium.

52% Relative Humidity

For the last 12 days, the climate chamber's relative humidity was reduced to 52 ± 3%. During the period the relative humidity of the air trapped inside the pile remained constant at 65%, as if the pile were sealed. The product moisture inside the pile remained relatively constant at the 0.17 to 0.23% level. The product moisture at the surface of the pile decreased rapidly from the 4.2 to 4.8% range to level out around 0.12% after 7 days.

Observations/Remarks

After the 42-day period, the probes were removed and the product was discarded after visual inspection. The following observations and remarks are worth mentioning:

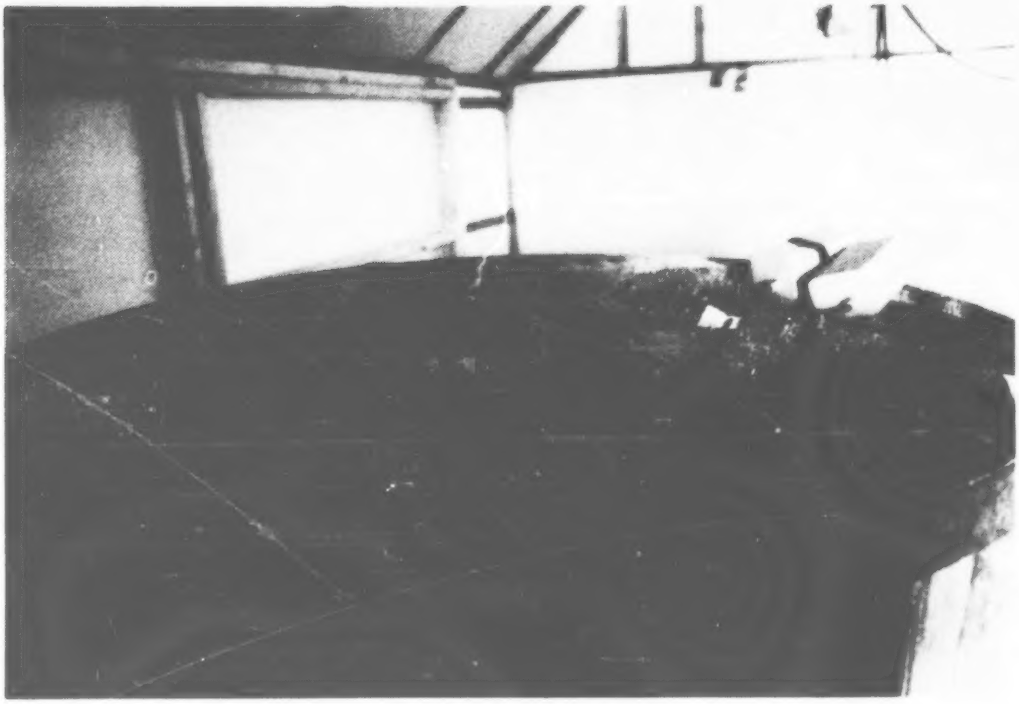
1. A significant amount of moisture is absorbed by the product, even below the critical relative humidity of KCl. At 72% relative humidity the product moisture levels out through the entire pile (core and surface) at about 0.10-0.12%, which is about three times higher than the initial condition. The fact that the moisture absorption levels out (reaches an equilibrium) at 0.10-0.12% suggests that the moisture is not in fact absorbed by the KCl but rather by the impurities (with a lower critical relative humidity level) located at the surface of the particles (surface contaminant).

2. The relative humidity level inside the pile never reached the climate chamber level, or even the critical RH of KCl, at any time during the experimental period. For example, at the climate chamber level of 87%, the relative humidity of the air inside the pile levelled out at 65%. At that level the product absorbs moisture without giving any sign of reaching an equilibrium. The effective critical relative

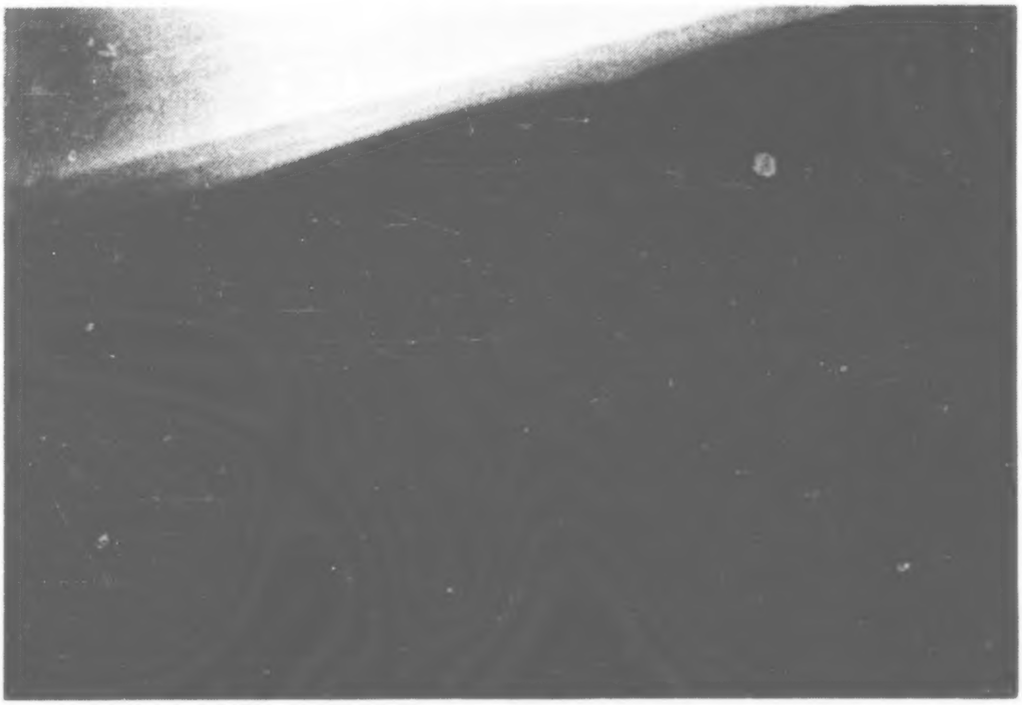
humidity of potash product seems to be lower than the theoretical critical relative humidity of potassium chloride. This suggests a liquid transport and absorption mechanism rather than a vapour transport as previously expected. Also, the pore structure of the stored product may effectively reduce the steady state humidity because of capillarity.

3. When the relative humidity was brought back to 52% during the last phase of the experiment, the product dried out at the top surface of the pile and all around the container wall, forming a crust about 1-2 cm thick. (See attached pictures.) This crust seemed to have trapped very efficiently the moisture and relative humidity inside the pile. Because the gradient of water vapour pressure within the pile was close to zero, there was no driving force for water vapour or liquid transport. Only at the pile surface where evaporation was occurring into the lower R.H. chamber air, did diffusion and water loss occur. This process also enhanced surface recrystallization of potash from condensed brine.

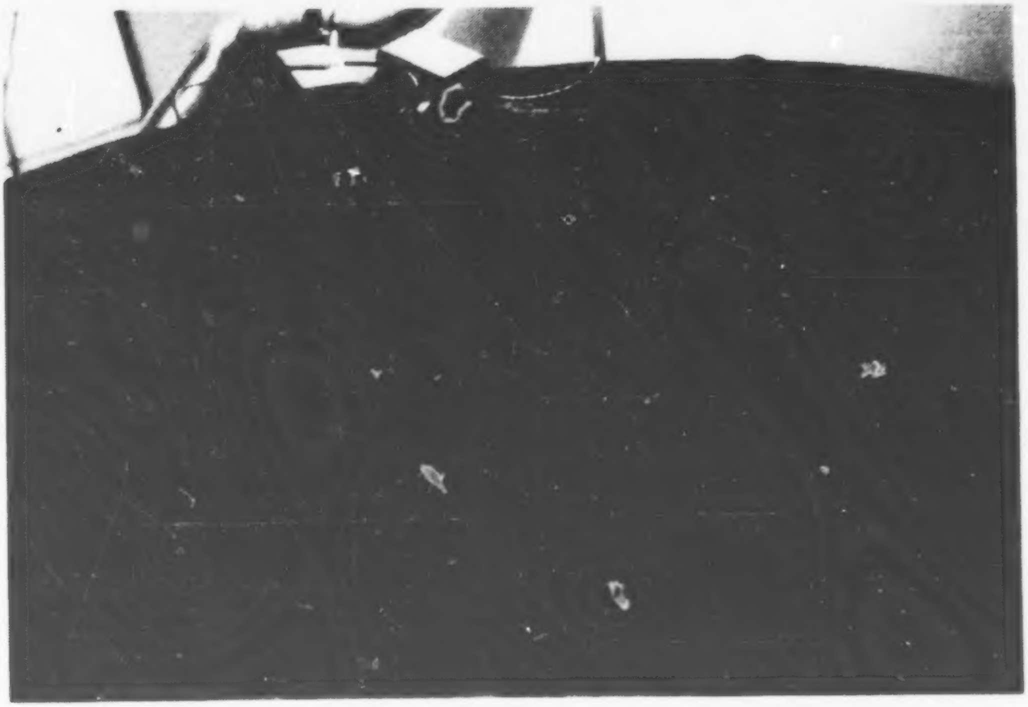
4. At the end of the experiment the product was starting to solidify and stand up pretty seriously. (See attached pictures.)



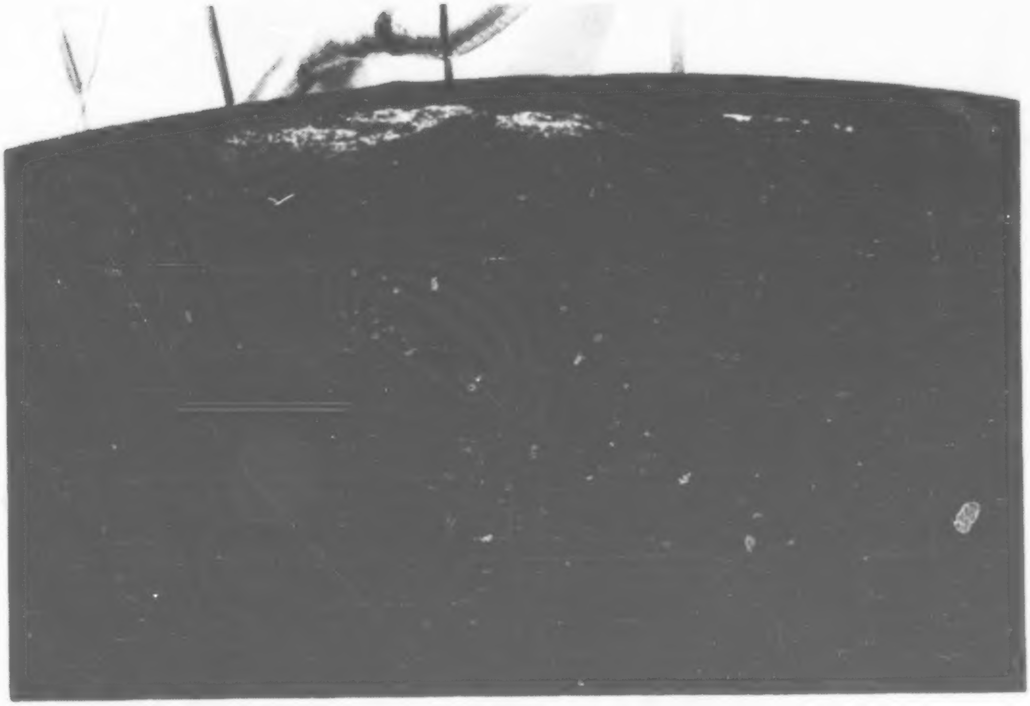
Vaisala HMP 135Y installation in product container.



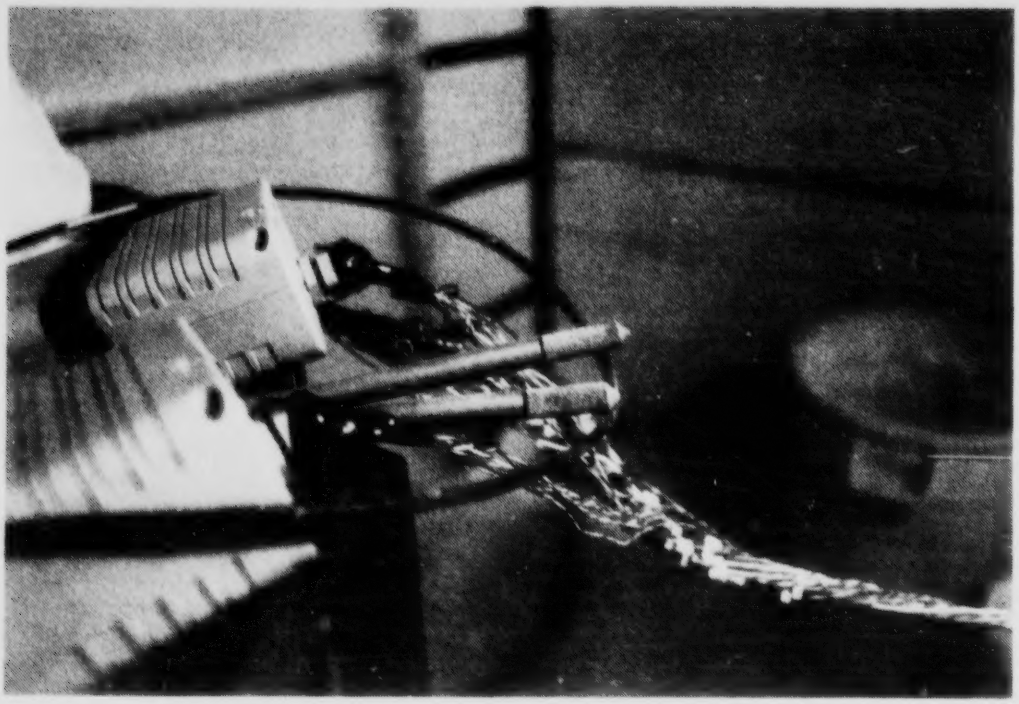
Crust formation along the wall of the product container
(After completion of the monitoring phase).



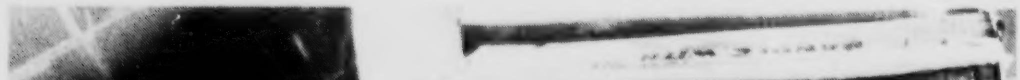
Probe recuperation after the monitoring phase.



Product consolidation after the monitoring phase.



Probe condition after removal from the product.



Sample of the crust formation around the container wall.

II SECOND MONITORING PERIOD

The second monitoring period lasted 66 days, during which the temperature was maintained constant at 27 ± 2°C. The relative humidity was changed periodically from 52 to 87%.

In this experiment, two product piles were monitored simultaneously. One consisted of Cory Granular 210, which is composed mainly of compacted particles. The other one was Cory Standard product, made primarily of natural crystals. Both product grades behaved similarly during the entire period.

57% Relative Humidity

The 57 ± 3% relative humidity level was maintained for 14 days. At the beginning, the product was in equilibrium with the pilot plant conditions, which were:

Temperature : 21.3°C
Relative humidity: 21%
Product Moisture : 0.067%

The chamber temperature was reached through the entire pile after 8 days of exposition. The RH set point of 57% was approached but not reached in either case. The product RH levelled off at 54%. The moisture level at the core of the pile increased by about 50%, to reach 0.10% H_2O, but progressively higher absorbed moisture levels were detected towards the surface.

75% Relative Humidity

When the RH set point was increased to 75%, the internal pile RH slowly augmented, to level off around 65%, which is significantly below the set point. The moisture level of 0.12% was reached at the core and 0.35% at the surface of the pile. These increases are very significant, considering that the chamber setting is well below the critical RH of KCl.

87% Relative Humidity

The chamber RH set point was then increased to 87 ± 3% for 6 days. During this time the pile's RH reached and levelled off around 72%, which is well below the chamber set point and still below the critical RH of KCl. Also, during this period extensive moisture condensation and migration occurred within each pile. Moisture levels exceeded 1% within the piles, about twenty times higher than normal, and were above 5% in the 1-2 cm outer layer of material.

52% Relative Humidity

For the next 11 days, the RH in the chamber was brought back down to 52%. The outer layer dried rapidly to 0.08-0.1% H_2O; however, the core moisture levels and RH remained exceptionally high, as if trapped inside the material.

85 - 52% Relative Humidity

For the last 21 days of the experiment, the products were submitted to another high-low RH cycle. The effects on the material, moisture absorption and RH levels were very similar to the first cycle.

Observations/Remarks

After the 62-day period, the probes were removed and the product was discarded after inspection. The following observations and remarks are important:

1. All the comments made for the first monitoring period are still valid, except that the moisture absorption and RH increases are more pronounced, as should be expected.

2. At the end of the experiment, the outer layer of each pile (2-3 cm) was caked solid and the core was standing up pretty well. The free flowing characteristic of the products was entirely inhibited.

The next section of the report describes the microscopic observations performed on samples retained at the end of the experiment.

MICROSCOPIC ANALYSIS

The microscopic analysis of the potash products that have been exposed to high-low relative humidity cycles confirmed several product behavioural concepts that were previously suspected but not demonstrated. Several photographs are presented in this section corroborating the general beliefs and real occurrences.

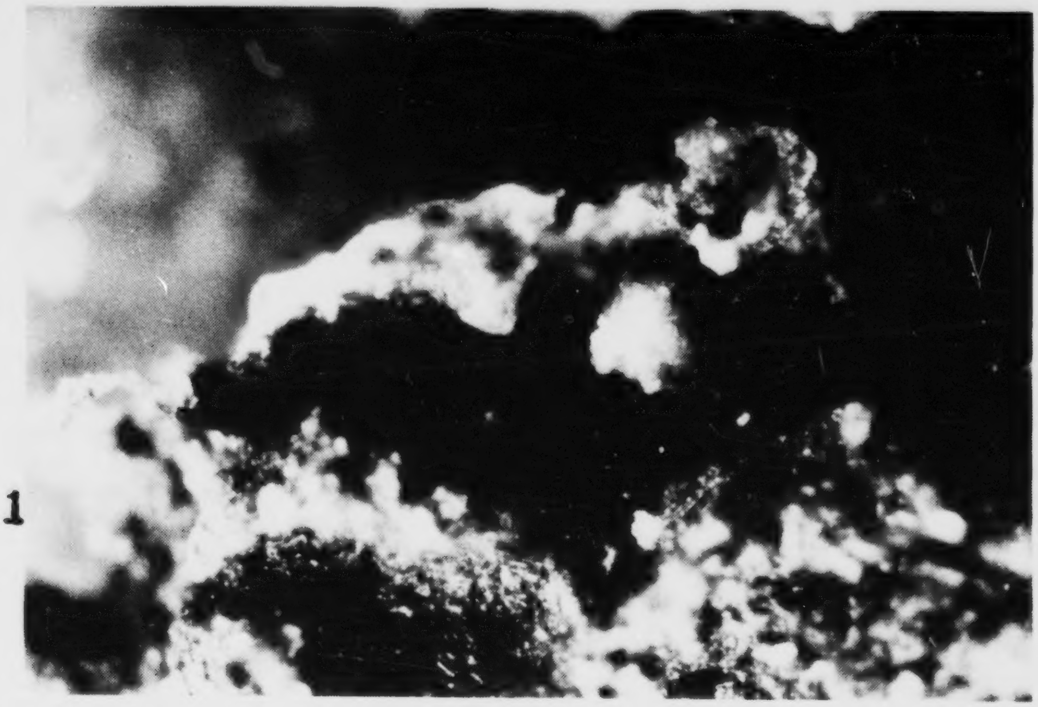
One very important behavioural concept that can be easily demonstrated is that compacted particles suffer a lot more extensive water damage than natural crystal exposed to the same conditions. Pictures 1 to 4 show some compacted Granular particles severely damaged by water absorption. Pictures 5 to 8 show some Standard product natural crystals exposed to exactly the same atmospheric conditions. In both cases, the products were exposed to moist and warm air (85-87% RH and 27°C) for a total of 13 days. The moisture level reached by both products was in the range of 5-6%. One should remember that these products were treated with dedusting and anticaking reagents in the same fashion as they are shipped from Cory Division which is typical for the industry.

As shown in pictures 9 to 16, three recrystallization possibilities prevail when the product dries up. One is that the KCl recrystallized at the surface of particles without touching any other grain. This recrystallization process is evident in pictures 9 to 12. Pictures 9 and 10 show large crystal growth on the surface of compacted Granular particles. The new crystals are fairly large, very clear, with a shiny liquid appearance. Pictures 11 and 12 show recrystallization of lot more, but smaller, crystals. The size of these new crystals is related to the amount of moisture present on the product just prior to drying. The product in pictures 9 and 10 was part of the outer layer of the pile, and the moisture content was as high as 5-6%. The moisture level on the product shown in pictures 11 and 12 was about 1-2%. These crystals are fragile and are likely to break away and degradate into fines and dust as soon as the product is handled.

It is also probable that recrystallization will occur at the contact point between particles. The newly formed crystals are bridging and cementing the particles at the contact points. Pictures No. 6 and 13 to 15 are showing such bridges. These bridges are inhibiting the flowing characteristics of potash material, thus inducing caking. The number of bridges is functional of the number of contact points between the particles while their size and, consequently, their solidity is related to the water availability in liquid or vapour forms.

The third possibility is KCl recrystallization in particle cavities and fractures, more or less restoring the original grains structures. The occurrence of this phenomenen seems to be very scarce, but is very well illustrated in picture No. 16.

1



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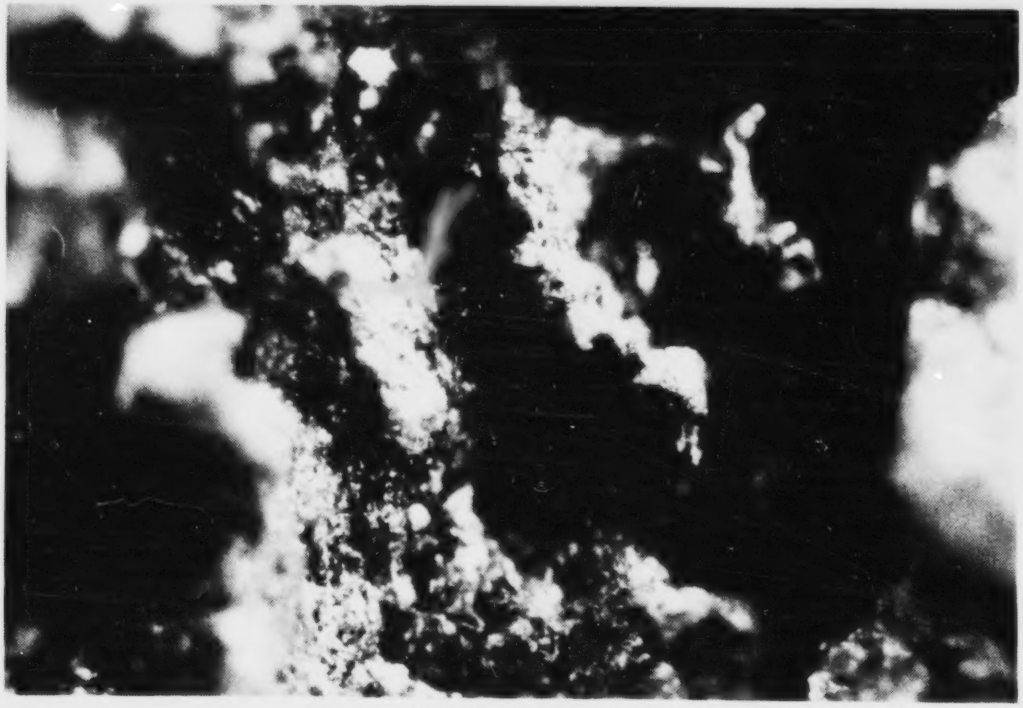
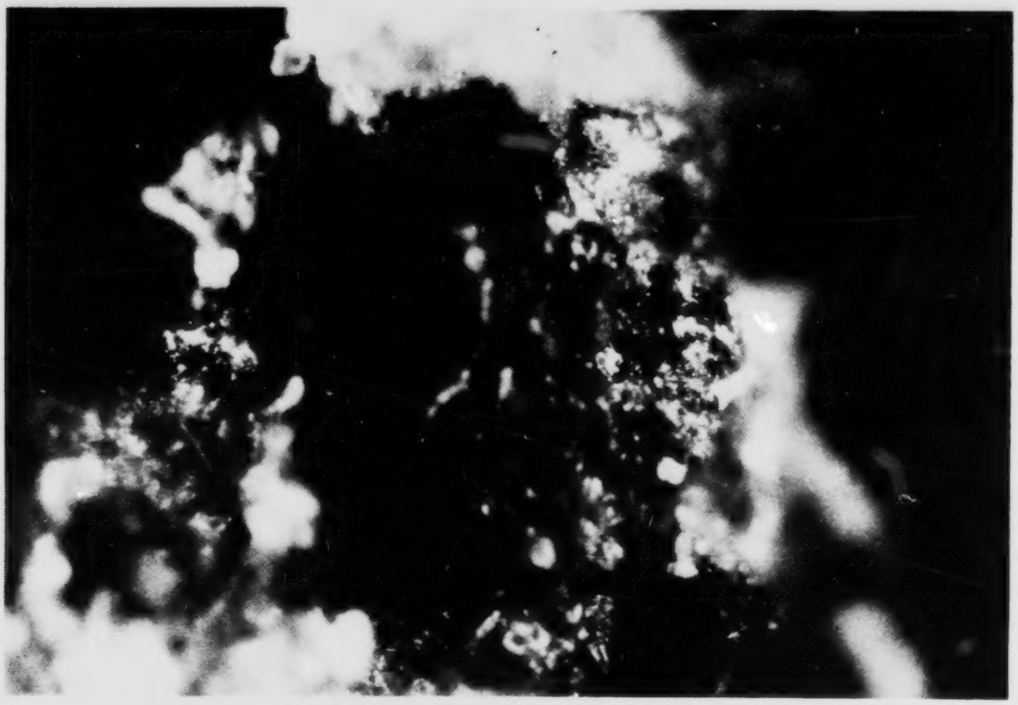


These pictures show compacted Granular particles damaged by water absorption. This water source was water vapour contained in moist and warm air (85-87% RH and 27°C). The total exposure time was 13 days. The moisture level reached by the product was in the 5-6% range.

3



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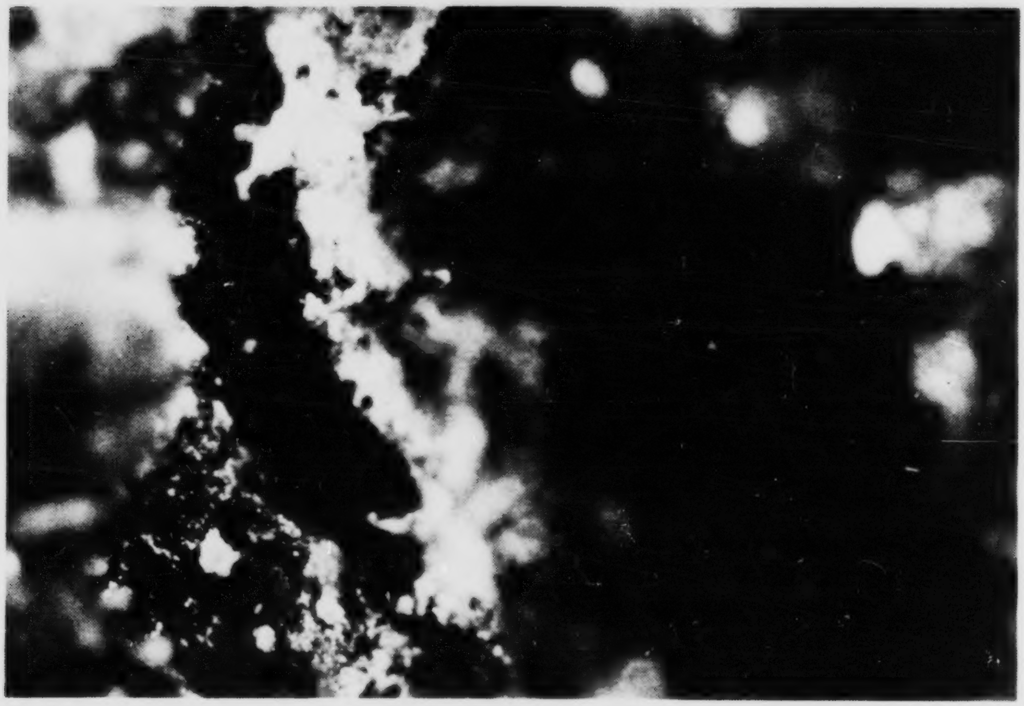
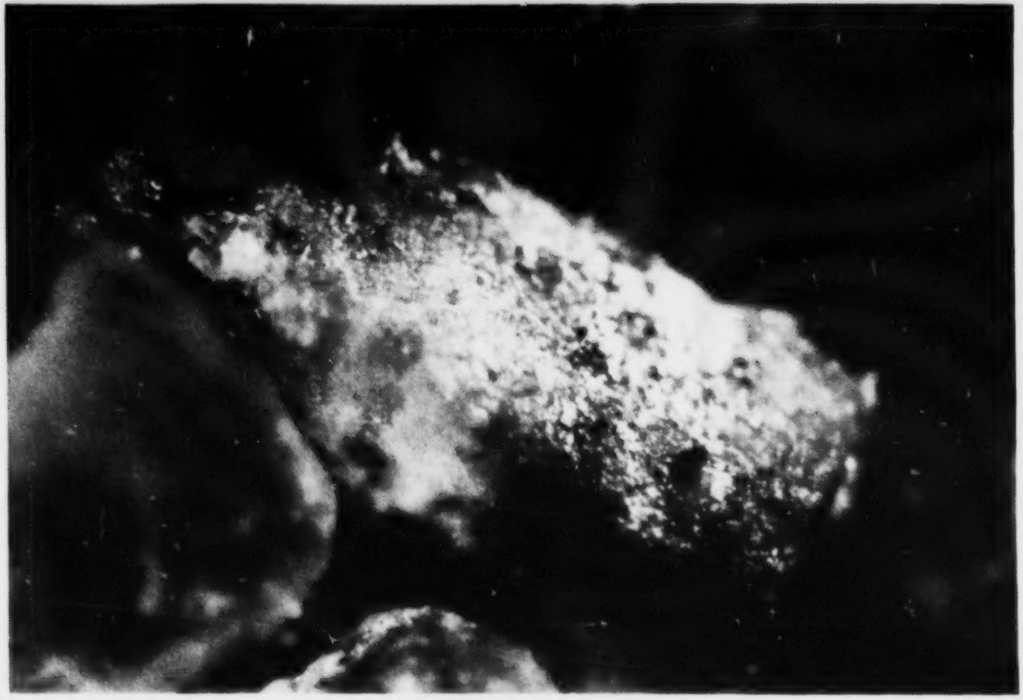
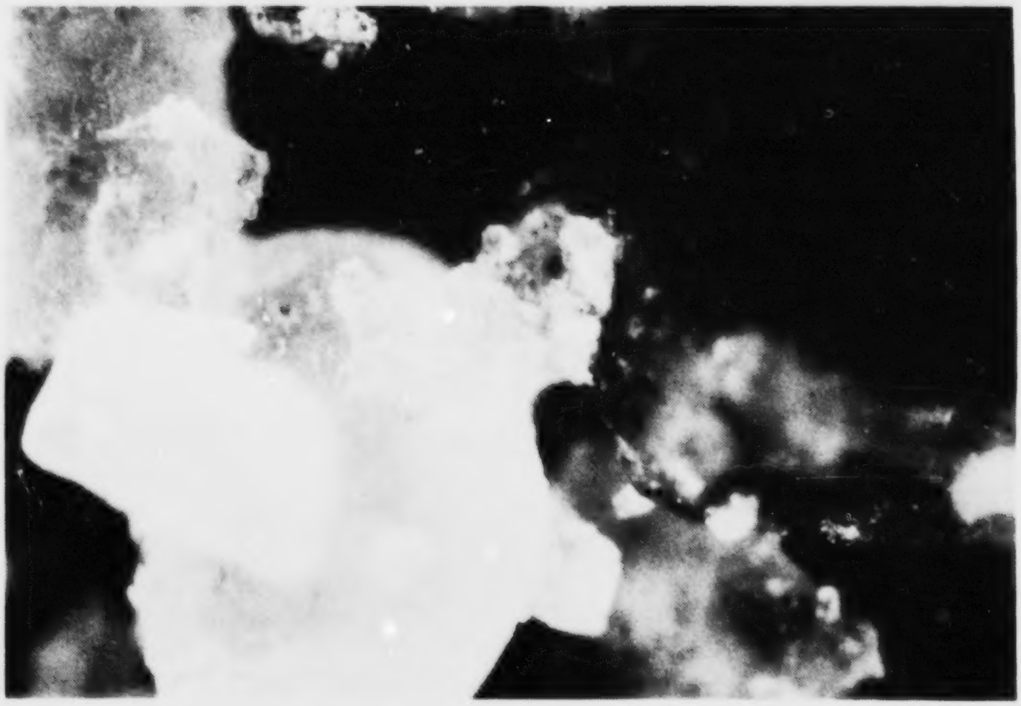


These pictures show compacted Granular particles damaged by water absorption. This water source was water vapour contained in moist and warm air (85-87% RH and 27°C). The total exposure time was 13 days. The moisture level reached by the product was in the 5-6% range.

5



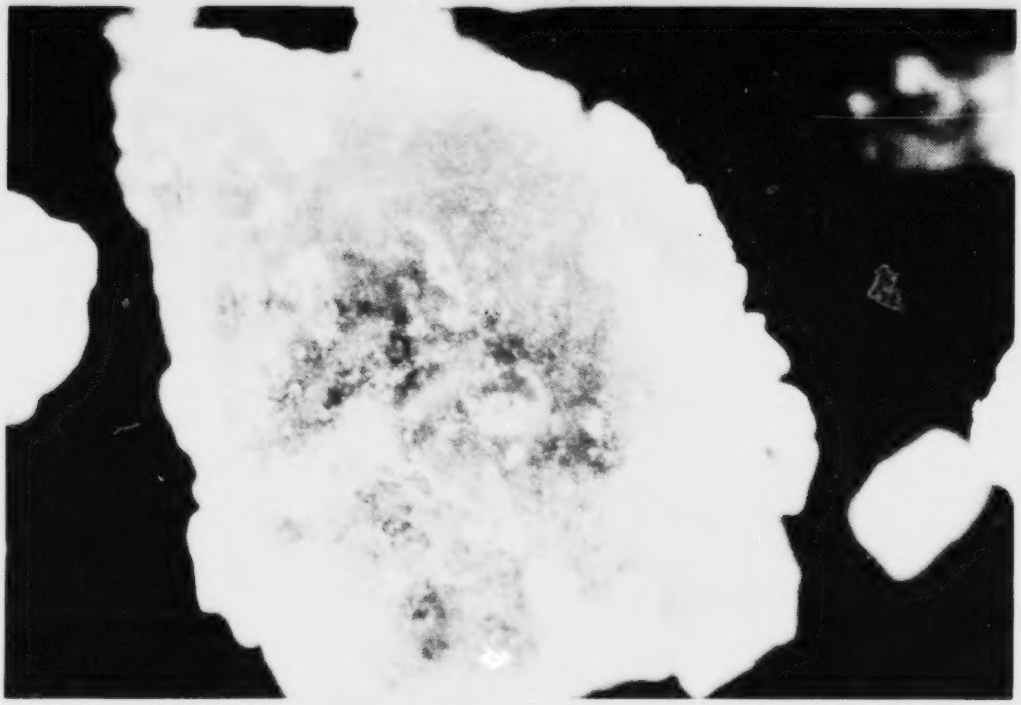
6



These pictures show Standard product natural crystal damaged by water absorption. The moisture level reached by the product was in the 5-6% range. The water source was water vapour contained in moist and warm air (85-87% RH and 27°C). The total exposure time was 13 days.

Picture 6 also shows the bridging of particles caused by water evaporation. This bridging is responsible for the product caking phenomena.

7



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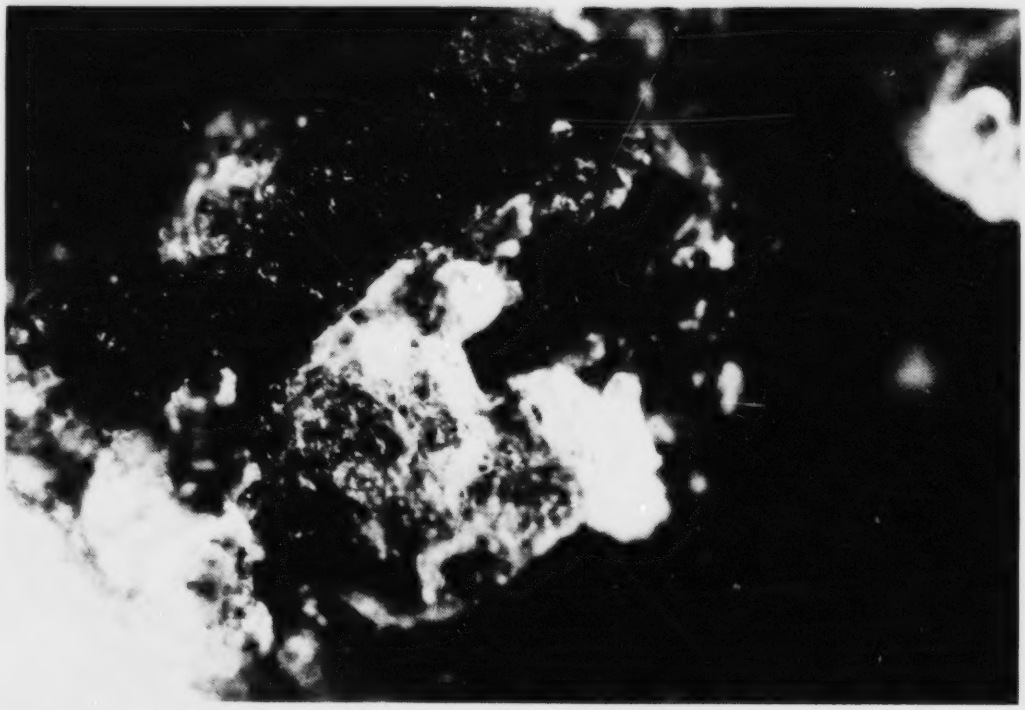
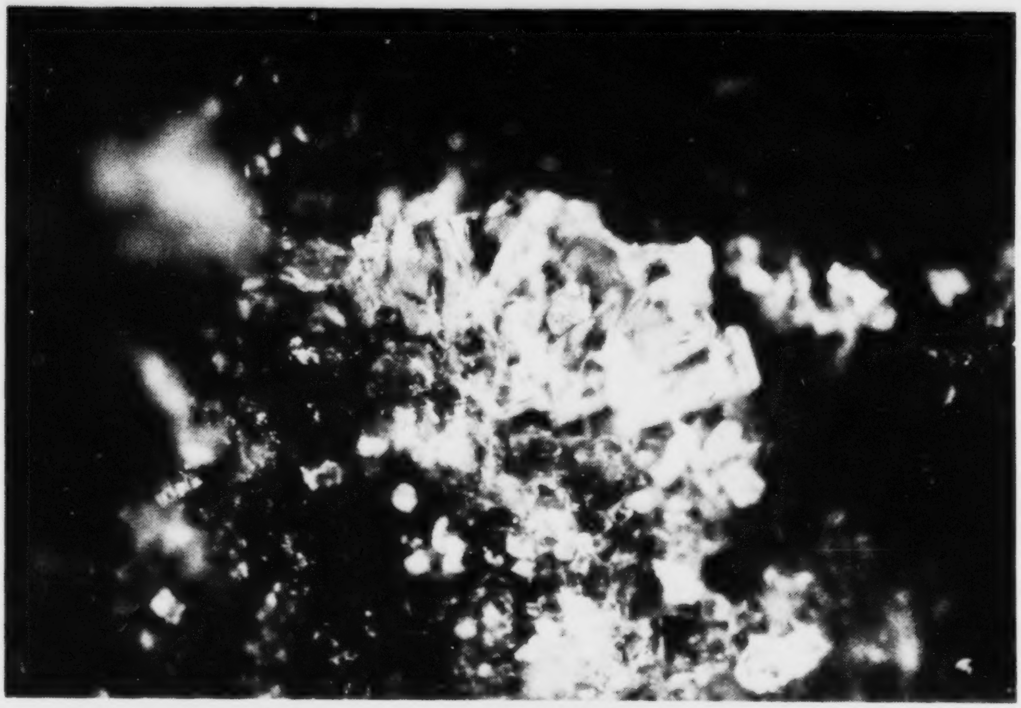


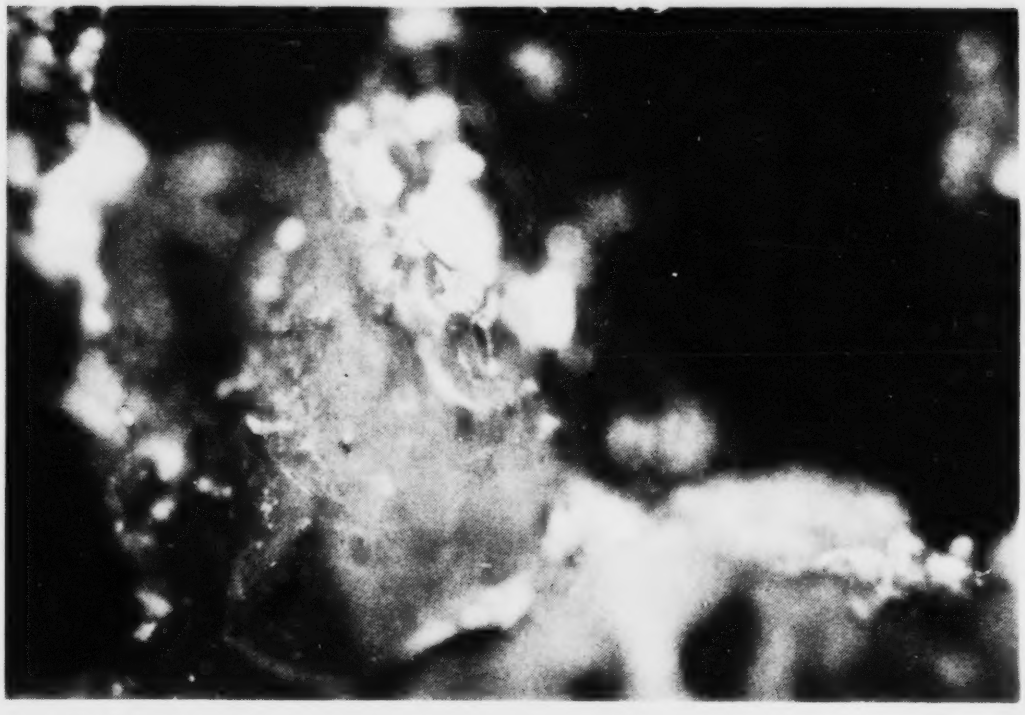
These pictures show Standard product natural crystals that have been exposed to moist and warm air (85-87% RH and 27°C) for a total of 13 days

Picture 7 shows fine particles recrystallized by water evaporation. In picture 8, the rectangular hole was likely formed by dissolution of an NaCl crystal, trapped in the KCl natural crystal.

9

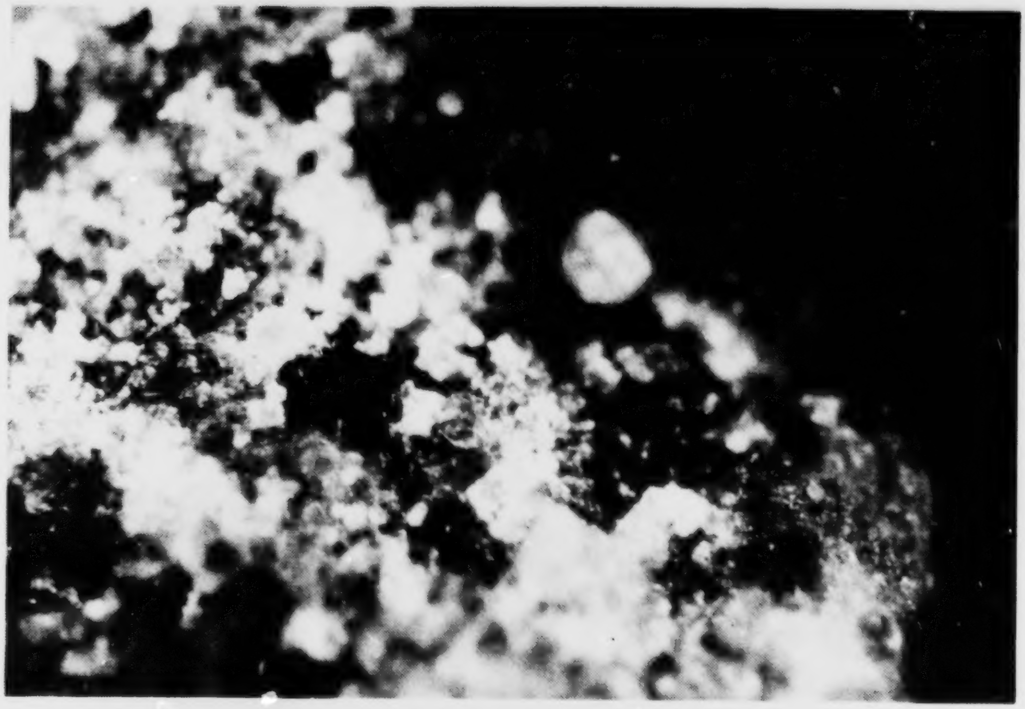


10

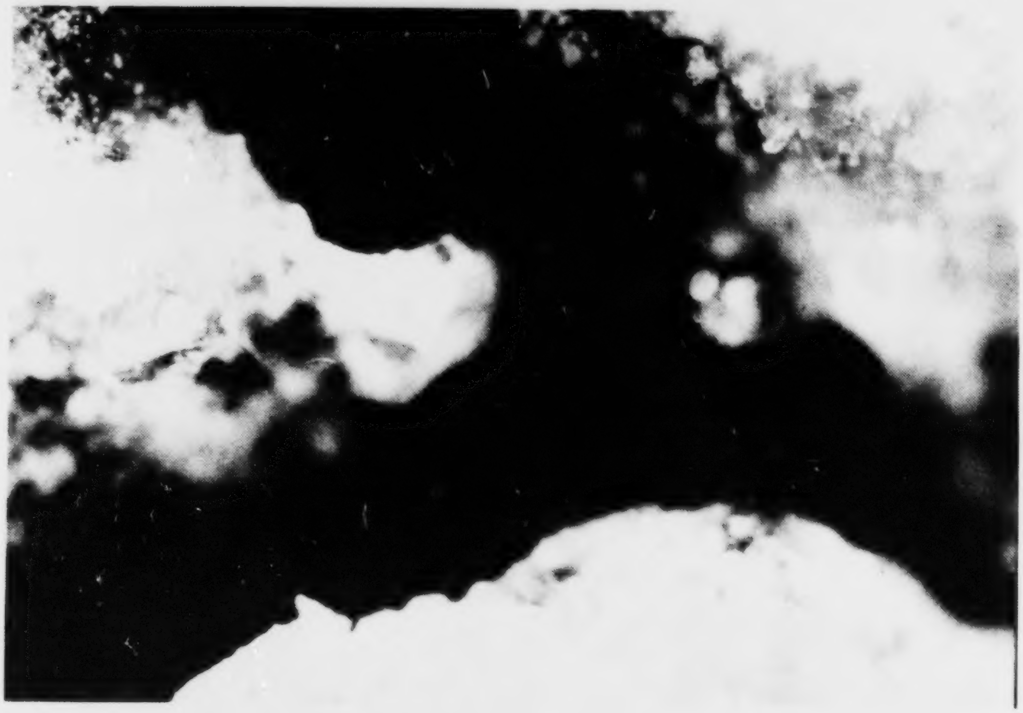


These pictures show evidence of KCl crystal growth on large compacted Granular particles. The product moisture was 5-6% just prior to drying and recrystallization.

11



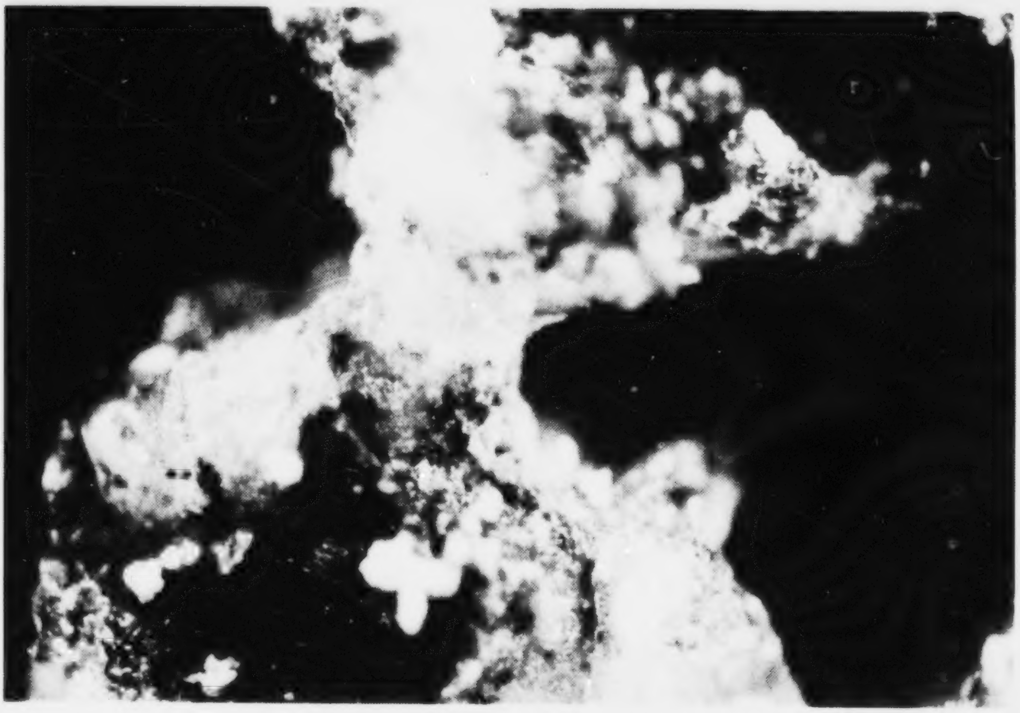
12



Again, KCl recrystallization on Granular product is evident. The product moisture prior to drying was about 1%. The crystals are smaller and are likely to generate considerable amounts of dust when handled.

13

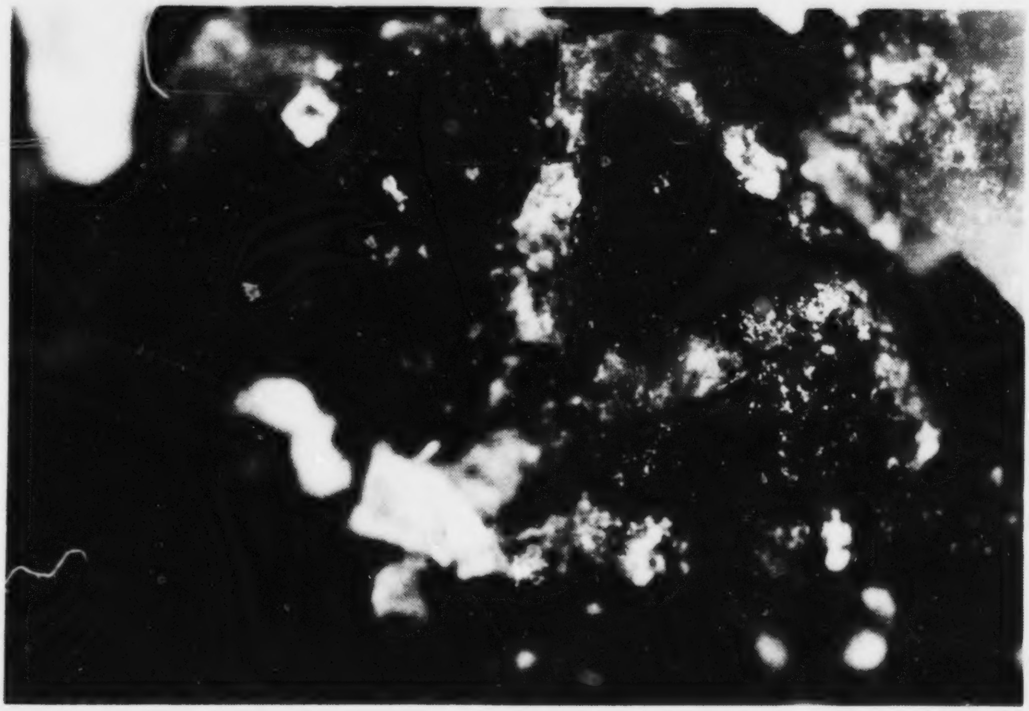
14



Pictures 13 to 15 are illustrating bridging and cementing of particles at their contact points. These bridges were observed on the caked material located in the outer layer of the monitored piled in which the moisture level reached 5-6% range.

15

16



In picture No. 16, grain cavities and fractures have been filled with secondary KCl recrystallization from brine produced in the water condensation process.

INTERPRETATION AND CONCLUSION

These experimental studies demonstrate that potash is a relatively fragile and moisture sensitive granular material and, that it perhaps should be managed as an unstable perishable product rather than as a stable mineral commodity. The downstream maintenance of product quality specifications that are rigorously met at the plant gate depends heavily upon the environmental or climatic conditions, the facilities and care in handling between the mine site and the customers.

The moisture condensation, absorption and transport mechanisms in potash particles or potash piles are very complex phenomena, and this project has explained only some of them. In fact, this study raises more questions than it answers and, therefore, it opens new avenues for research.

At the Potash Particles Level

Atmospheric moisture begins to condensate on the potash material at a atmospheric relative humidity level well below the theoretical critical relative humidity of potassium chloride which is 83%. In the second phase of exposure, the moisture level in the outer layer of stored potash material increased from 0.07% to 0.14% at a R.H. setting of 52%. At 72% R.H., the moisture level raised to 0.35%. In both cases an equilibrium appeared to have been reached. At the 87% R.H. setting, the moisture condensation increased rapidly to 5 - 8% within 6 days without giving any sign of equilibrium. One theory explaining this phenomenon is that the potash particles surface contaminants such as sodium, magnesium and calcium salts have individual critical relative humidity lower than the potassium chloride and are, therefore, dissolved the first ones in a specific order. An apparent equilibrium or steady state condition is reached at each R.H. level because each contaminant is present in a limited supply. If this is the case, the surface brine created by water condensation and surface contaminant dissolution would have a different composition at each R.H. level.

This would also explain why the moisture damage is a lot more pronounced on the compacted particles than the natural crystals. This is because each individual grain composing a compacted particle is itself covered by surface contaminants, and that after compaction the contaminants end up in the core of the particle. The condensed moisture is infiltrating the particle and dissolving the surface contaminants of each individual grain by the capillaries and porosities that exist at the grain boundaries.

These two theories explaining these occurrences seems to be logical but, in fact, neither one of them have been proven or demonstrated by our experiments.

At the Potash Piles Level

Qualitatively the data gathered indicate that absorbed or condensed moisture may migrate by either a vapour phase or a surface liquid film mechanism or both, depending on the product location within the pile and on the amount of moisture in contact with the product.

The potash product located in the outer layer of the pile (the first 1 - 2 cm) absorbs a large amount of moisture (the limit is probably at the total dissolution of the material) as soon as the R.H. in the climate chamber reaches the critical R.H. of the potassium chloride. In this case, the moisture condensation can only be due to a vapour absorption mechanism. However, the variables (forces or factors) influencing this mechanism and the reaction kinetics are unknown at this stage.

In the case of the pile, the moisture absorption and transport appears to be more related to surface liquid film mechanism. This mechanism implies a liquid moisture migration toward the centre of the pile, rather than a vapour phase absorption or condensation on the product. This seems to be the case because the critical R.H. level is never reached in the core of the pile and that the moisture level obtained is significantly higher (300 - 400% higher) than the level reached by the outer material in equivalent R.H. conditions. This moisture migration also implies that the brine created would have a different composition at different locations in the pile because the surface contaminants dissolved on the outer particles, as explained previously, would also migrate towards the centre, perhaps driven by capillarity. Again, the variables (forces or factors) and rates influencing this mechanism and its kenetic are unknown.

A vapour transport mechanism in the outer layer of material and a liquid transport mechanism in the core of the pile suggests a transition zone in which both mechanisms would be present. These experiments permitted the identification of these three different zones but their delimitation have not been achieved.

For an academic research point of view, the development of a mathematical model predicting product behaviour in hot humid environments can be very challenging, but will still require a few more years of research. A simulation model idea may be very attractive but its application in the field might prove to be very difficult due to rapid and unpredictable changes in atmospheric conditions.

However, in the interim, short term actions can be taken to minimize product deterioration and degradation due to moisture contact. Some can be described as follows:

1. Removal of particle surface contaminants to minimize water absorption and condensation below the critical relative humidity of potassium chloride.

2. Investigate the possibility of reducing porosity and capillary in compacted particles (i.e. identifying the relationship between porosity capillary and compactor roll pressure, and binding agents, including water or brine).

3. Conduct moisture absorption, impact and shear stress tests on compacted particles, natural crystals, crystallized grains and compacted crystallized particles separately to get a better knowledge of their individual properties and be able (at least theoretically if not practically) to achieve an optimum particles blend in granular and coarse products.

Adopting the view that potash is a perishable commodity helps to formulate plans to manage product quality from the production site to customer. The following is some field research items that will assist the protection of potash product:

1. Investigate the possibility of shipping warm products from the mine sites. Heat will hinder the moisture absorption considerably. This project should include a heat transport analysis on loaded railcars and, if required, a feasibility study on replacing existing car liners with heat resistant lining material.

2. For long term storage in a humid environment, investigate the possibility of insulating the product from the moisture source, even if the product is stored inside a building. This could include covering the product with tarpaulin or with biodegradable water repellant chemical agents (foam or spray).

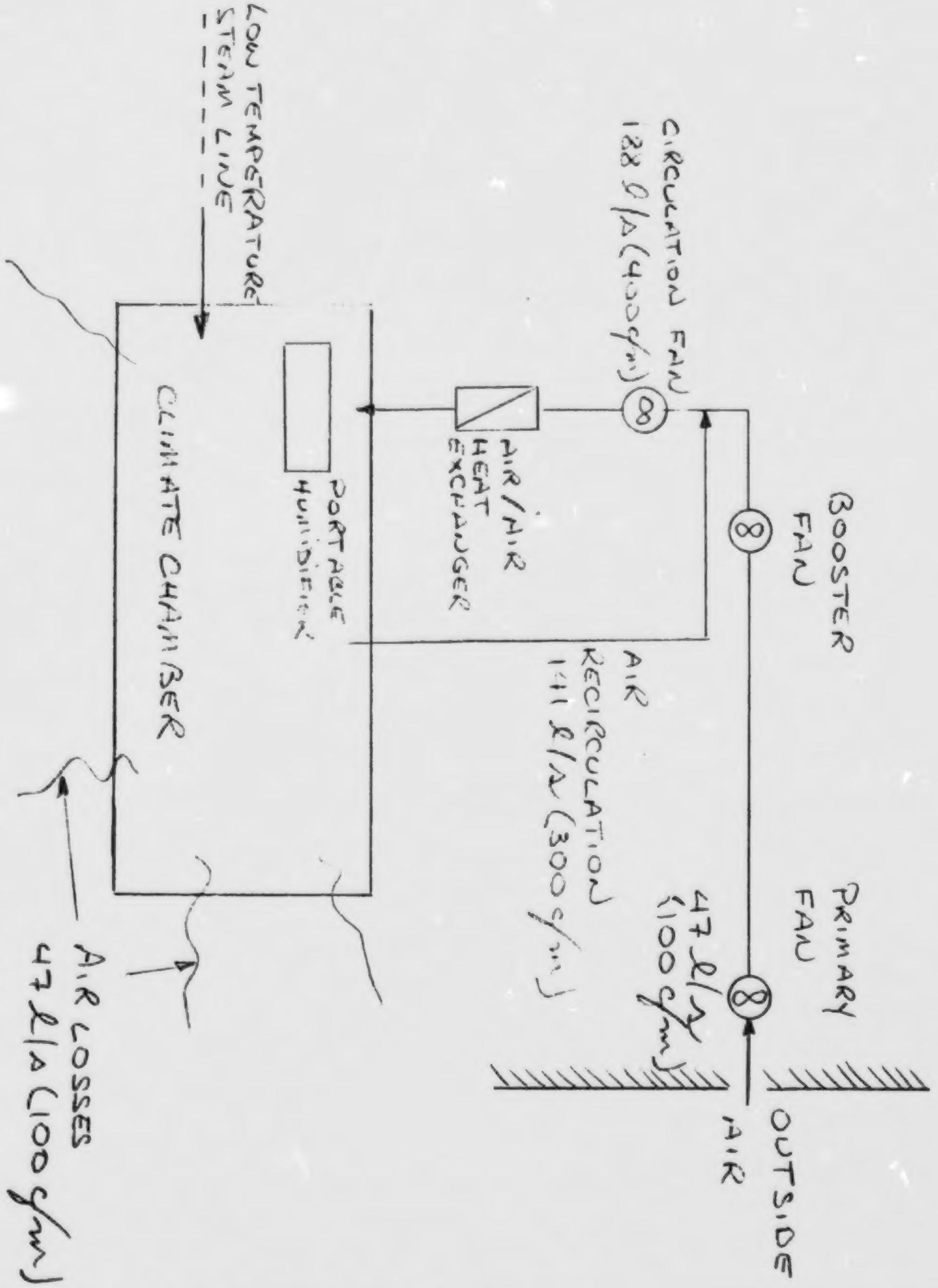
The above discussion demonstrates the complexity of "shelf life" factors affecting potash material and the importance to protect it from abuse. Rethinking of attention to the details in handling, shipping and warehousing is becoming a priority to be able to compete effectively and efficiently in an always more complex market with more demanding customers.

APPENDIX I

CLIMATE CHAMBER INSTALLATION

AND PRODUCT HANDLING

FIGURE No.1



CIRCULATION FAN
128 l/s (4000 cfm)
LOW TEMPERATURE STEAM LINE
CLIMATE CHAMBER
PORTABLE HUMIDIFIER
AIR/AIR HEAT EXCHANGER
BOOSTER FAN
AIR RECIRCULATION
141 l/s (300 cfm)
PRIMARY FAN
47 l/s (100 cfm)
OUTSIDE AIR
AIR LOSSES
47 l/s (100 cfm)

PCS Mining TECHNICAL SERVICES

PROJECT 1224.02
EQUIP. No. CLIMATE CHAMBER
SUBJECT
BY D.G.
DATE APRIL 1988
PAGE OF
FILE No.

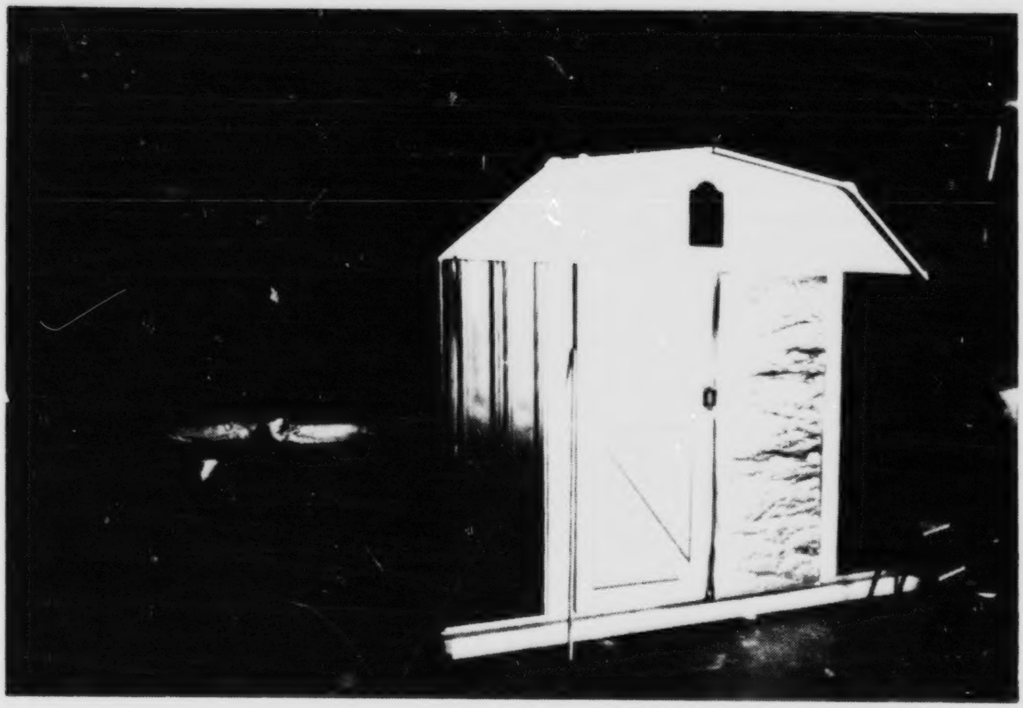


Figure 1. Climate chamber

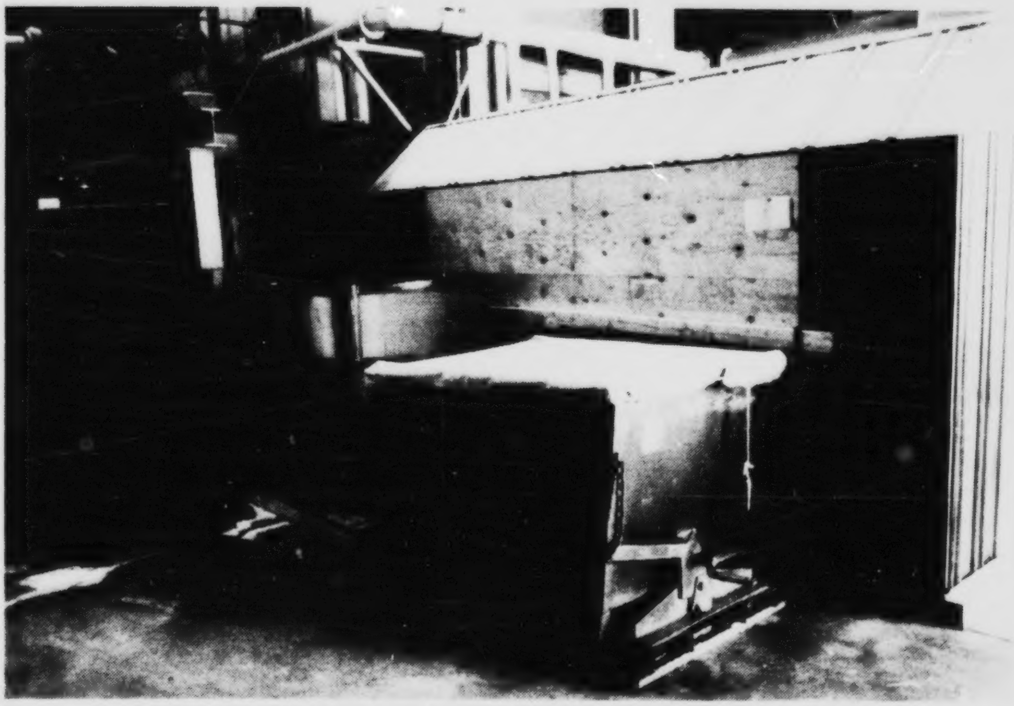


Figure 2. Climate chamber and product container.

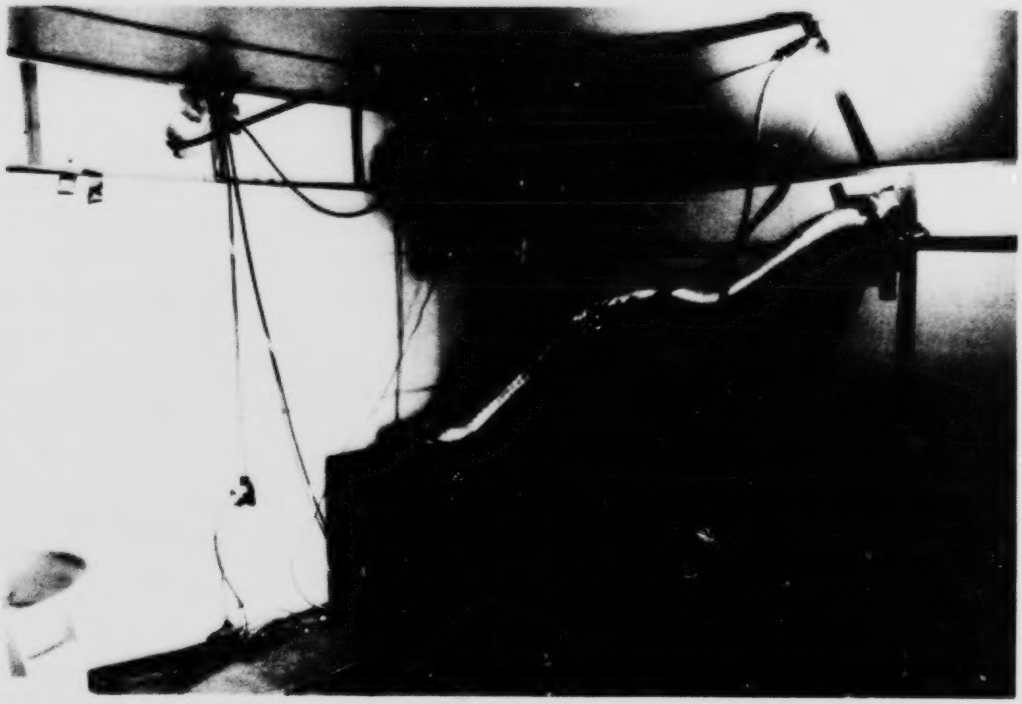


Figure 3. Climate chamber interior

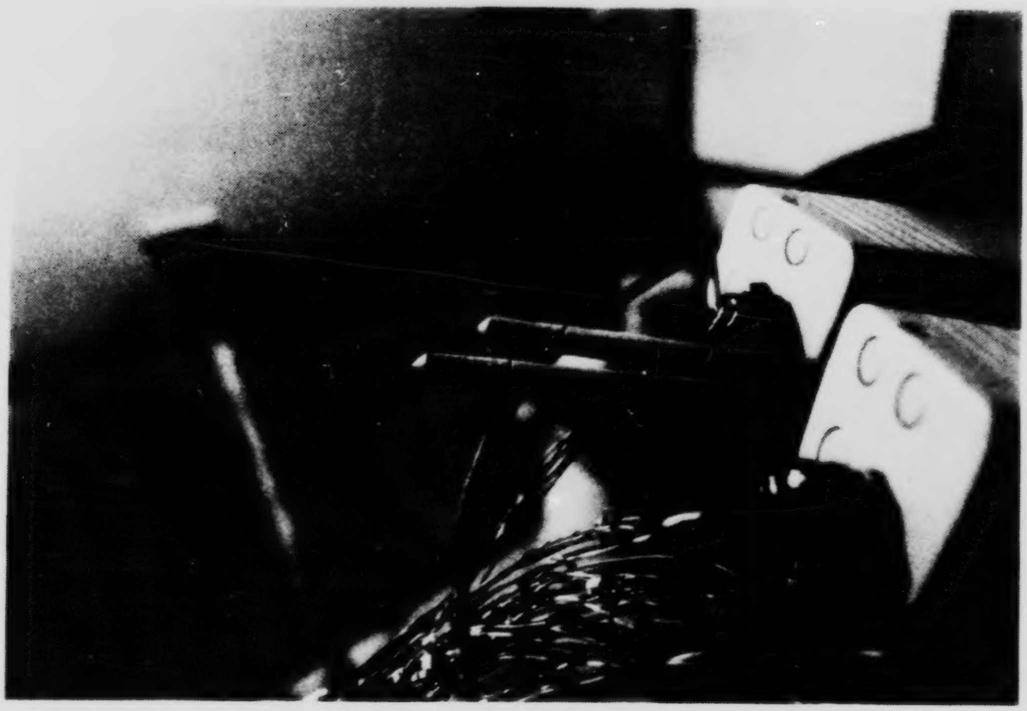


Figure 4. Vaisala HMP 135Y probes and transmitter.

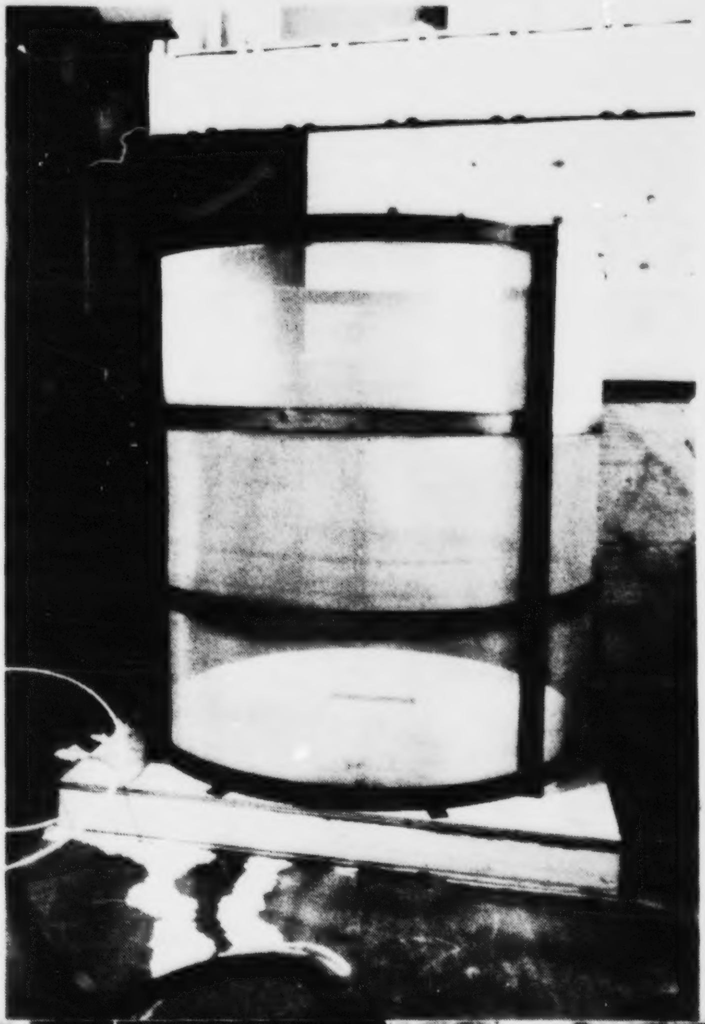
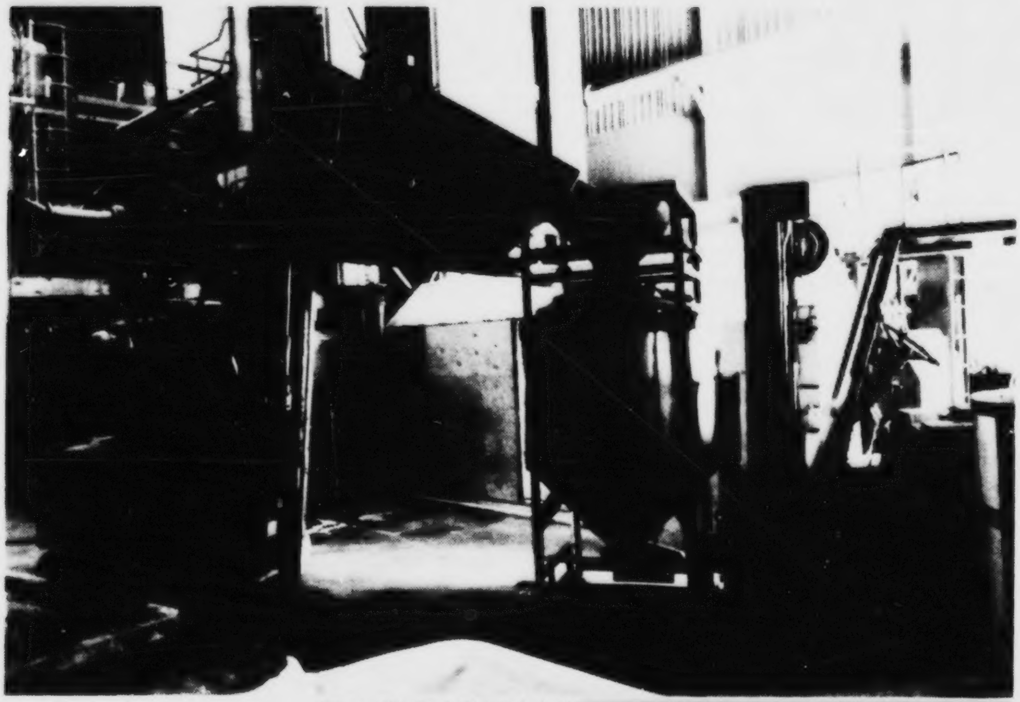


Figure 5.
Product container
(built in stainless steel 20 mesh screen cloth)

Figure 6.
Soltech computer
and data logger
Acro 900 system



Product transfer into
a product bin and into
the permeable container

APPENDIX II

CONDUCTIVITY PROBE EVALUATION

Potash
Corporation
Saskatchewan

MEMORANDUM

To G. Strathdee

Date January 15, 1988

From D. Gauthier

Re CONDUCTIVITY PROBE EVALUATION (SPPA PILE MONITORING PROJECT)

Professor de Jung's conductivity probe (Wenner arrangement, Figure #1) was tested and evaluated at the PCS Pilot Plant. The objective was to correlate conductivity measurements with moisture levels in potash products. The testing procedure used is shown in Exhibit #1.

From the testing it was discovered that the conductivity probe cannot be used indiscriminately with all potash products (see Exhibit #2). In the moisture range observed (0.02 to 0.14%) no significant correlation was found between the conductivity measurements and the moisture level (correlation coefficient r = 0.17). Also, no significant correlation was discovered between the conductivity and the product size (represented by the SGN number). In this case, the correlation coefficient r was calculated at -0.29.

However, using each product separately, some correlation has been discovered. To discover this correlation, higher moisture levels were required (0.02 to 0.40% range). Unfortanetly, the regression equations differ very significantly with change in the bulk density (loose and packed densities) and particle size distribution (see Exhibit # 3 & 4).

The reading variation with product bulk density alone, can be as high as one order of magnitude. The degree of variation inferred by these two additional variables is so great that the correlation equations cannot be used for the intended purpose in the SPPA Potash Pile Monitoring project, because the product bulk density and product size distribution cannot be estimated throughout the pile due to various packing degree and segregation.

After this wide measurement variation was discovered with Rocanville Granular and Standard products, the testing was discontinued. Another approach will have to be used to measure insitu the product moisture level.

DANIEL GAUTHIER
Process Engineer

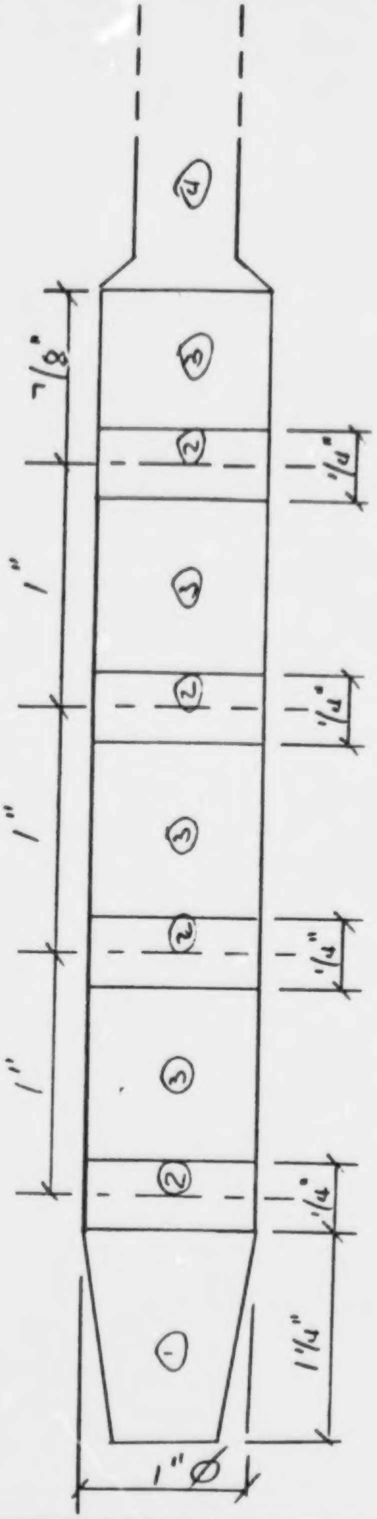
c.c. J.A. Gosselin, Research Chemist, Kalium Chemicals

DG/cc
ID-3646K

FIGURE No1.

CONDUCTIVITY PROBE CONFIGURATION

(WENNER ARRANGEMENT.)


④
③
7/8"
②
1/4"
③
1"
②
1/4"
③
1"
②
1/4"
③
1"
②
1/4"
①
1 1/4"
1" Ø

① NYLON PENETRATION HEAD
② COPPER ELECTRODES (4)
③ PLEXIGLAS INSULATORS
④ ALUMINUM HANDLE

PCS Mining ENGINEERING DEPARTMENT

PROJECT POTASH PILE MONITORING	
EQUIP No	
SUBJECT	
BY	DATE
PAGE OF	FILE No

EXHIBIT # 1

CONDUCTIVITY PROBE EVALUATION TESTING PROCEDURE

Objective: Discover correlation between conductivity measurements and moisture level, partical size and reagent level.

1. Pour about 1 kilogram of product of known characteristic, such as:
   - size distribution
   - SGN
   - treatment level (kg/tonne)
   - Moisture level (%)

   in the mixing pot, and blend carefully for 5 minutes.
2. Gently transfer the blended sample in about 1 litre plastic bottle* containing the conductivity probe.
3. Take about 30 grams sample for moisture analysis (gravimetric method).
4. Measure conductivity of the material using the probe.
5. Pack the material in the bottle as much as possible by shaking it and knocking it gently onto the floor and refill the bottle.
6. Re-measure conductivity of the material.
7. Transfer back the sample into the mixing pot and moisturize uniformly (rotate pot for 5 minutes).
8. Repeat items 2 to 7 for 4 different moisture levels from about 0.02 to 0.4%.
9. Repeat items 1 to 8 with the following products:

   Rocanville Granular : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Rocanville Coarse : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Rocanville Standard : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Rocanville Sp. Standard: Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Cory Granular : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Cory Sp. Standard : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Allan Coarse : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Allan Standard : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Lanigan Granular : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Lanigan Standard : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Kalium Granular : Untreated and Treated (0.5 kg/t and 1.0 kg/t)
   Kalium Standard : Untreated and Treated (0.5 kg/t and 1.0 kg/t)

* Several containers of different volumes were pre-tested to ensure that the container itself does not interfere with the conductivity measurement.

EXHIBIT # 2

CONDUCTIVITY MEASUREMENTS

PRODUCT (NOT TREATED)	SGN	CONDUCTIVITY	MOISTURE %
Rocanville Granular	255	0.117	0.044
Rocanville Coarse	195	0.113	0.111
Rocanville Standard	80	0.114	0.071
Rocanville Sp. Standard	30	0.109	0.138
Cory Granular	220	0.104	0.022
Cory Sp. Standard	25	0.111	0.048
Allan Coarse	235	0.110	0.021
Allan Standard	80	0.114	0.036
Lanigan Granular	235	0.100	0.019
Lanigan Standard	100	0.101	0.033
Kalium Granular	230	0.105	0.010
Kalium Standard	75	0.125	0.025

Regression:

I Conductivity $= a + b$ (moisture %)

$= 0.1087 + 0.0314$ (moisture)

$r = 0.1744$

$r^2 = 0.0304$ $\sigma y = 0.007$

II Conductivity $= a + b$ (SGN)

$= 0.1137 - 2.3436 \times 10^{-5}$ (SGN)

$r = -0.2919$

$r^2 = 0.0852$ $\sigma y = 0.007$

EXHIBIT # 3

ROCANVILLE GRANULAR SGN 255 (NOT TREATED)

CONDUCTIVITY		MOISTURE
LOOSE MATERIAL	PACKED MATERIAL	%
	0.117	0.044
	2.97	0.135
	2.33	0.234
	2.54	0.297
	2.10	0.356
	0.115	0.151
	2.45	0.203
2.640	2.30	0.140
0.217	1.30	0.087
0.111	0.126	0.066

Regression:

I (Packed Material) Conductivity $= a_1 + b_1$ (moisture %)
$= 0.4966 + 6.6444$ (moisture %)
$r = 0.5965$, $r^2 = 0.3558$
$\sigma_y = 1.126$

II (Loose Material) Conductivity $= a_2 + b_2$ (moisture %)
$= -2.5675 + 36.4189$ (moisture %)
$r = 0.9709$, $r^2 = 0.9426$
$\sigma_y = 1.431$

III (Combined Material) Conductivity $= a_3 + b_3$ (moisture %)
$= 0.2980 + 7.6980$ (moisture %)
$r = 0.6217$, $r^2 = 0.3865$
$\sigma_y = 1.17$

EXHIBIT # 4

ROCANVILLE STANDARD SGN 80 (NOT TREATED)

CONDUCTIVITY LOOSE MATERIAL	PACKED MATERIAL	MOISTURE %
	0.114	0.071
2.72	2.40	0.201
1.47	1.75	0.113
0.113	0.111	0.092

I (Packed Material) Conductivity $= a_1 + b_1$ (moisture %)
$= -1.0584 + 18.0477$ (moisture %)
$r = 0.8861, \quad r^2 = 0.7851$
$\sigma y = 1.16$

II (Loose Material) Conductivity $= a_2 + b_2$ (moisture %)
$= -1.4163 + 21.0635$ (moisture %)
$r = 0.9342, \quad r^2 = 0.8727$
$\sigma y = 1.30$

III (Combined Material) Conductivity $= a_3 + b_3$ (moisture %)
$= -1.1975 + 19.3215$ (moisture %)
$r = 0.9081, \quad r^2 = 0.8248$
$\sigma y = 1.13$

APPENDIX III

INVESTIGATION OF MOISTURE MEASUREMENT IN PROCESSED POTASH USING MICROWAVE TECHNIQUES

r

Potash Corporation of Saskatchewan

MEMORANDUM

To: G.Strathdee

Date: 1988, September 12

From: D.Gauthier

Re: THE MEASUREMENT OF MOISTURE CONTENT IN PROCESSED POTASH USING MICROWAVE TECHNIQUES

Attached to this memo you will find a copy of Dr.S.Kumar's main and supplementary reports on the above mentioned topic. Dr.Kumar is with the Centre of Communication Studies, Department of Electrical Engineering, University of Saskatchewan (Appendix I and II).

The object of the study was to evaluate the possibility of measuring the moisture level in potash material using microwave techniques. These techniques included an investigation of the dielectric constants, (dialectric losses), and resonant effects. The products used in the study were untreated Granular 210 and Standard products from Lanigan Division. The experiments were designed for moisture levels ranging from 0.035% to 0.450% H_2O.

Dr. Kumar has identified a positive correlation between the moisture level (in percent) and the dielectric loss in each potash material. Frequencies in the 400-800 Mhz range appear to be more suitable for moisture measurement because the percentage change in the reading is greater than the percentage change of moisture (steeper slope). However, the correlation functions for the Granular 210 and the Standard products are significantly different, indicating that other variables are influencing the relationship. One of the variables identified by Dr. Kumar is the bulk density of the product. I strongly suspect that the reagent level and type (dedusting and anticaking reagents) are other variables that are affecting the relationship. However, the reagent effects have not been studied at this stage.

No resonant effect has been identified in the potash material for the frequency range under consideration, from 50 Mhz to 4000 Mhz.

Dr. Kumar has concluded that the attenuation constant (dielectric loss) of the potash is a function of moisture content, frequency, property of potash and potash grade (bulk density). He has also identified two avenues for future work:

a) The establishment of an empirical formula correlating the dielectric loss with the moisture content for a given frequency and bulk density. Standard curve fitting methods are available to establish this relationship. More data (experiments) are required to achieve this objective with a good degree of confidence.

b) The other area is to explore the feasibility of developing a "radar" type instrument to measure remotely the moisture content in potash material.

At this stage of development, the usefulness of these two proposed research alternatives is entirely academic or scientific. From the practical or engineering point of view these developments are of very little value for the following reasons:

1. The correlation discovered between the moisture content and the dielectric loss of the products is not as straightforward as it seems to be. The dielectric loss is correlated with total moisture; occluded and surface. The moisture levels indicated in the report refer only to surface moisture.

   As long as the relationship (if there is one) between surface and occluded moisture is not better understood, the relationship between the dielectric loss and the surface moisture level will be an unreliable approximation only (too wide standard deviation). This is because the level of occluded moisture varies widely in the compacted particles, natural crystals and crystallized product, and the fact that the occluded moisture can be three to four times higher than the surface moisture. This could probably explain the measurement errors mentioned in the report, for the Granular product, in the 0.1 to 0.2% moisture range. The relatively wide standard deviation calculated in the linear regression analysis (Appendix III) can also be caused by this same variable.

   Therefore, for a reliable correlation, an additional variable must be included in the model. This variable has probably been overlooked because all the experiment measurements have been done using the same product samples.

2. Two other variables affecting the dielectric loss measurements are the bulk density and particle size distribution of the product. The linear regression analysis demonstrates that the slope and the intercept of the Granular and Standard product functions are too different (in magnitude) to be neglected.

   Therefore, the natural particle segregation occurring in a product pile will make a "radar" type instrument absolutely useless as long as the particle size distribution and degree of packing is not incorporated in the model. This is assuming that a reliable segregation model is already available, which is definitely not the case.

3. The type and quantity of treatment reagents (dedusting and anticaking) are strongly suspected to be variables also influencing the model, but this relationship has not been studied.

Therefore, the development of a useful empirical model requires a lot more work than previously anticipated. The function between the moisture content and the dielectric loss is a complex multivariate relationship requiring the development of at least three sub-models:

i) Relationship between occluded and surface moisture.
ii) Particle segregation model in large pile.
iii) Product bulk density in relation to particle size distribution, height of the pile, internal pressure, and time.

Also the "radar" type instrument would be of limited use because only pile surface measurement would be achievable. In-depth measurement, without disturbing the product in place, would have far greater value.

In light of all the above factors, it is evident that the probability of a breakthrough, without spending a lot of resources (time, manpower, and money) is very slim. Therefore, it is not recommended at this time to pursue the research alternatives proposed by Dr.S.Kumar.

Daniel Gauthier

Daniel Gauthier,
Process Engineer

Attachments

cc D.K.Storer
R.Lacroix (no attachments)

ns

APPENDIX I

THE MEASUREMENT OF THE MOISTURE CONTENT IN PROCESSED POTASH USING MICROWAGE TECHNIQUES

(Main Report)

by

Centre of Communication Studies
Department of Electrical Engineering
University of Saskatchewan

S. Kumar
S.Pan
G.Harron

UNIVERSITY OF SASKATCHEWAN
CENTRE FOR COMMUNICATION STUDIES

DEPARTMENT OF
ELECTRICAL ENGINEERING

SASKATOON CANADA
S7N 0W0

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Process Engineer
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Suite 500, 122 - 1st Avenue South
Saskatoon, Saskatchewan
S7K 7G3

Centre for Communication Studies
Department of Electrical Engineering
University of Saskatchewan
Saskatoon, Saskatchewan
S7N 0W0
(306) 966 5394
FAX (306) 343 6669

August 3, 1988

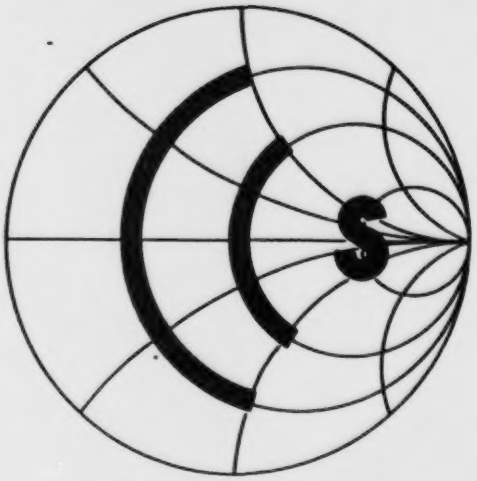
Dear Daniel:

Enclosed are the original and one copy of the report investigating "The Measurement of the Moisture Content in Processed Potash using Microwave Techniques". We will leave it up to you to distribute them to the necessary people. We want to thank you for you support through the investigation. The project was interesting in that it applies microwave technology to the physics and chemistry of a material. If you have any questions feel free to contact myself or Surinder.

Thankyou

G. Harron

Gerald Harron
Research Officer



CENTRE FOR COMMUNICATION STUDIES

DEPT. OF ELECTRICAL ENGINEERING
UNIVERSITY OF SASKATCHEWAN
SASKATOON, SASK.
CANADA

The Measurement of the Moisture Content in Processed Potash using Microwave Techniques

by:
Potash Corporation of Saskatchewan

and

Centre for Communication Studies
Department of Electrical Engineering
University of Saskatchewan

Dr. Surinder Kumar
S. Pan
Gerald Harron

July 27, 1988

The Measurement of the Moisture Content in Processed Potash using Microwave Techniques

(A Research Report to PCS Mining Corp.)

According to the investigation outline in the proposal for this project, the search work has been done as reported as follows:

a) A microwave system to measure the moisture content in processed potash has been established. The system is of the experimental grade and it is illustrated in figure 1. In the system, the HP8510 network analyzer is used to measure the S-parameters, S11(w) and S21(w), of the sample holder filled by the potash. Measurement calibration is performed using the standards in the HP85050A 7mm calibration kit. The full 2-port calibration with synthesized step sweep mode and 256 point averaging is used. The S-parameters are measured at 401 frequency points which are uniformly sampled from 50 Mhz to 4000 Mhz. Conversion from S-parameters data measured by the HP8510B to the dielectric properties is accomplished by reading trace data into the computer, performing the required conversions, and then plotting the results.

The sample holder is a coaxial transmission line. The physical dimensions of the sample holder is shown in figure 2. The characteristic impedance of the holder with air is 173.55 ohms.

The basic theory for the computation of complex permittivity (εr) and permeability (Ur) from S-parameter data is described as follows. For the sample material installed in the 173.55 ohm coaxial tranmission line shown in figure 3, the S-parameter is related to the reflection coefficient (Γ) between Zs and Zo when the lenght of material is infinite as shown in equation (1) while it is related to the transmission coefficient (τ) in the materials with lenght of d as shown in equation (2). The complex permittivity (εr) and permeability (μr) are related to Γ and τ by equation (3) and (4). These four equations can then be used to compute εr and μr from S11 and S21. Note that the limitation $|\tau| \leq 1$ is used to get the unique proper solution in computation and the unique solution is guaranteed for the predetermined frequency range and holder lenght.

b) The phase constant (ε') and attenuation constant (ε''), as the real part and imaginary part of the complex permittivity respectivity, have been measured for 2 grades of processed potash and 8 levels of moisture content for each grade. The moisture content range under consideration is from 0.03% to 0.7%. This range is suitable to the real case. The moisture content was measured by weighing the measured potash and the dried one. The original measurement results along with detailed measurement conditions are shown in the appendix of the report. The results indicate that the phase constant is not sensitive to the moisture content but the attenuation constant is apparently related to the moisture content.

The relationships between the measured attenuation constant and the moisture content at 3 frequencies (400 Mhz, 800 Mhz, and 2000 Mhz) are shown in Table 1 and Table 2. It has been observed that higher sensitivity appears at the lower frequency band.

c) No resonant frequency has been found in potash for the frequency range under consideration, from 50 Mhz to 4000 Mhz.

d) The measurement results show that the moisture content is the main determining factor of the attenuation constant while the potash grade has an effect to the attenuation constant but less important. When the moisture content increases, the attenuation constant increases. When the size of potash increases (bulk density decreases), the attenuation constant decreases. The resolution of the measurement in the development system is about 0.05%. This means that the system can not correctly recognize the moisture content change smaller than 0.05%. The attenuation constant of potash is a function of moisture content, frequency, property of potash and potash grade (bulk density). This relationship may be represented approximately by an empirical equation but more measurement data is required to determine the equation.

e) The investigation also provides some information about microwave heating of processed potash. The microwave heating is mainly characterized by the effective loss factor $\varepsilon e''$, given by

$$\varepsilon e'' = \varepsilon'' + \sigma/(2\pi f \varepsilon o)$$

where ε'' is the attenuation constant of the material
σ is the dielectric conductivity of the material
f is the microwave frequency
$\varepsilon o = 10^{-9}/(36\pi)$ (farad/meter)

Too high a value of the dielectric loss will result in a small skin depth, which annuls the desirable volumetric heating effect. On the other hand, too low a dielectric renders the material practically transparent to the incoming energy. Experience has shown that materials with a effective loss factor in the range $2 < \varepsilon e'' < 100$ will be suitable for processing with this form of energy. Therefore a knowledge of the dielectric properties is very important when assessing the feasibility of heating a given material. The measurements of the attenuation constant of processed potash give us this knowledge.

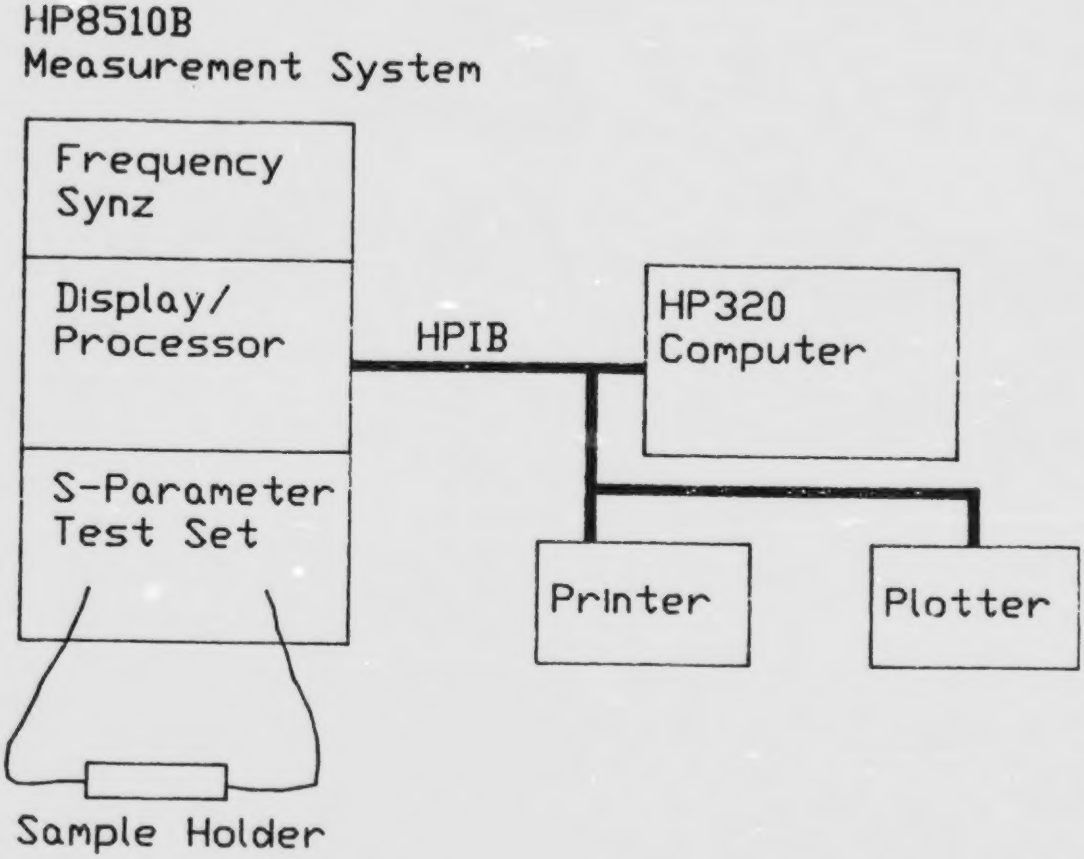

HP8510B
Measurement System
Frequency
Synz
Display/
Processor
S-Parameter
Test Set
Sample Holder
HPIB
HP320
Computer
Printer
Plotter

Figure 1
Illustration of the Microwave System

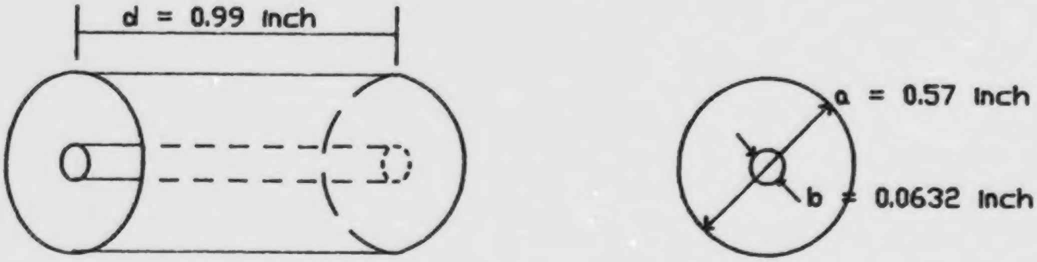

d = 0.99 inch
a = 0.57 inch
b = 0.0632 inch

Figure 2
Physical Dimensions of the Sample Holder

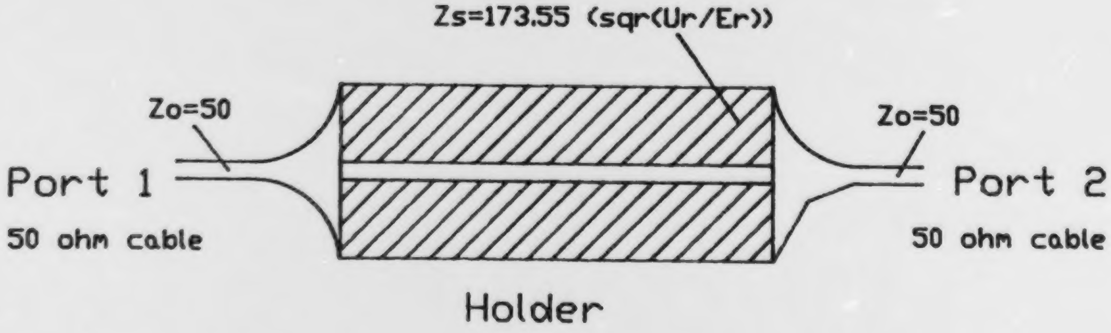

Zs=173.55 (sqr(Ur/Er))
Zo=50
Port 1
50 ohm cable
Zo=50
Port 2
50 ohm cable
Holder

Figure 3

Illustration of Sample Holder with filled material

$$S11(w) = \frac{(1-T^2)\Gamma}{1-T^2\Gamma^2} \qquad (1)$$

$$S21(w) = \frac{(1-\Gamma^2)T}{1-T^2\Gamma^2} \qquad (2)$$

$$\Gamma = \frac{Zs-Zo}{Zs+Zo} = \frac{173.55\sqrt{\frac{Ur}{Er}} - 50}{173.55\sqrt{\frac{Ur}{Er}} + 50} \qquad (3)$$

$$T = \exp\left[-j\,\frac{w}{c}\,d\sqrt{UrEr}\right] \qquad (4)$$

c = speed of light

$$Er = Er' - j\,Er'' \qquad (5)$$

Moisture Freq	0.03588%	0.1226%	0.1301%	0.1496%	0.1719%	0.2780%	0.3304%	0.4486%
400 Mhz	0.083	0.42	0.58	0.58	0.50	1.0	1.25	1.67
800 Mhz	0.33	0.58	0.67	0.58	0.54	0.98	1.17	1.50
2000 Mhz	0.92	1.17	1.18	1.08	0.98	1.42	1.67	1.75

TABLE 1

The Attenuation Constant Measurements for Processed Potash
In Grade A

Moisture Freq	0.0377%	0.113%	0.240%	0.297%	0.366%	0.411%	0.572%	0.679%
400 Mhz	0.083	0.83	1.30	1.66	2.08	3.25	4.04	4.50
800 Mhz	0.42	0.99	1.33	1.50	1.66	2.5	2.98	3.25
2000 Mhz	1.25	1.75	1.58	1.75	1.75	2.25	2.5	2.66

TABLE 2

The Attenuation Constant Measurements for Processed Potash
In Grade B

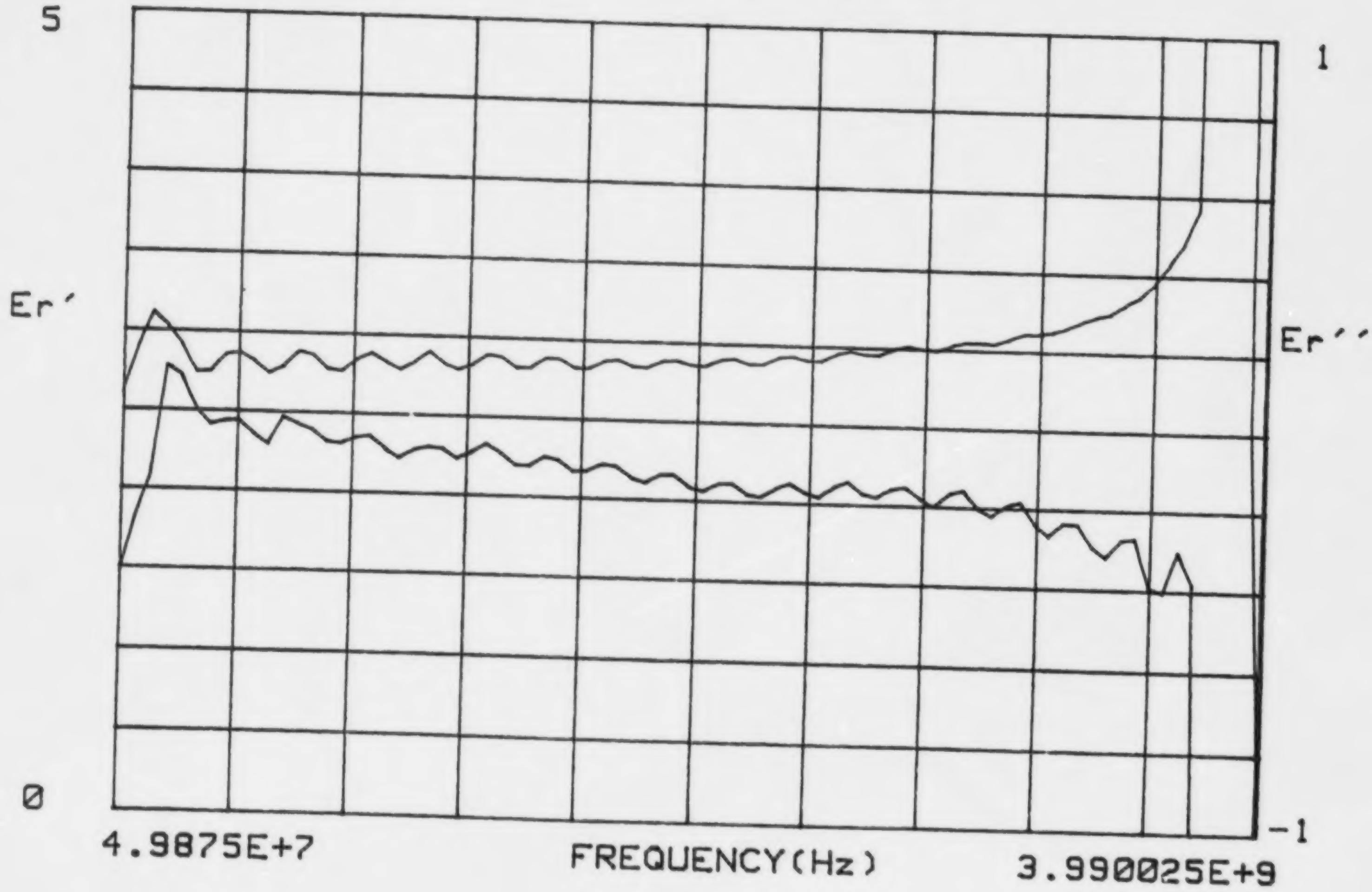
APPENDIX

```
OPERATING PARAMETER     Channel 1          Channel 2

NUMBER of POINTS         401                401
SWEEP TIME               100.0 ms           100.0 ms
SOURCE POWER             10.0 dBm           10.0 dBm
POWER SLOPE              0.0 dB/GHz         0.0 dB/GHz
                         OFF                OFF
PORT 1 ATTEN.            0.0 dB             0.0 dB
PORT 2 ATTEN.            0.0 dB             0.0 dB

ELECTRICAL DELAY         0.0 s              0.0 s
PHASE OFFSET             0.0 °              0.0 °
MAGNITUDE SLOPE          0.0 dB/GHz         0.0 dB/GHz
MAGNITUDE OFFSET         0.0 dB             0.0 dB
IF AVERAGING FACTOR      256.0              256.0
                         ON                 ON
SMOOTHING APERTURE       0.1 % SPAN         0.0 % SPAN
                         OFF                OFF
```

```
OPERATING PARAMETER     Channel 1            Channel 2

PORT 1 EXTENSION         97.69999 ps          97.69999 ps
PORT 2 EXTENSION         34.57699 ps          34.57699 ps
Z0                       50.0 Ω               50.0 Ω
CAL TYPE                2-PORT               2-PORT
                         ON                   ON
GATE START              -65.398 ps           -2.275 ps
GATE STOP                231.6 ps             294.723 ps
WINDOW                  NORMAL               NORMAL
GATE SHAPE              MINIMUM              NORMAL
                         OFF                  OFF
MARKER 1                 89.775559 MHz        89.775559 MHz
MARKER 2                 4.0 GHz              4.0 GHz
MARKER 3                 4.0 GHz              4.0 GHz
MARKER 4                 4.0 GHz              4.0 GHz
MARKER 5                 4.0 GHz              4.0 GHz
```

09:29:28 18 Jun 1988

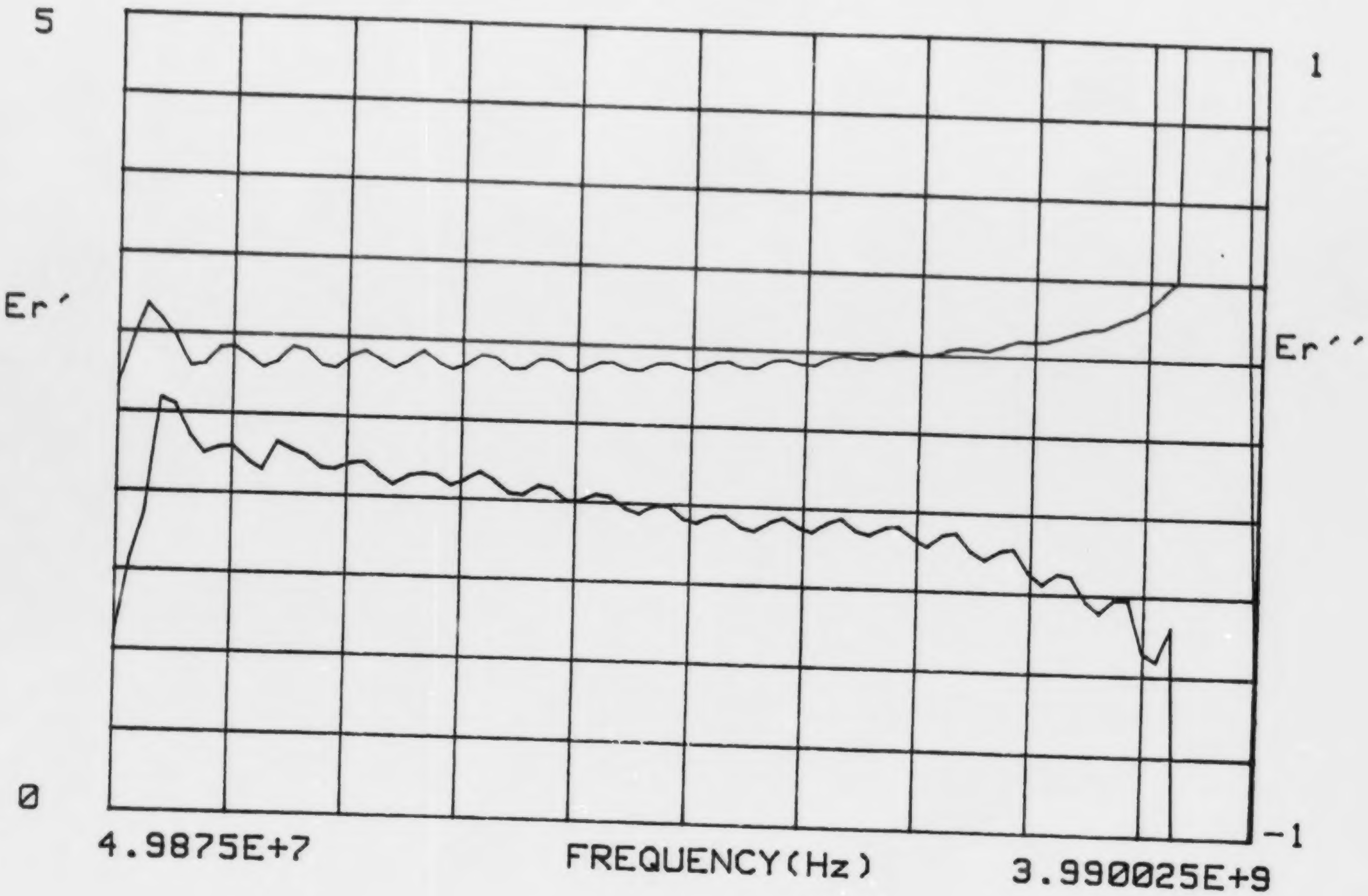
original d 0.03588% moisture



Grade A , 0.03588% moisture

09:37:28 18 Jun 1988

0.1226% moisture

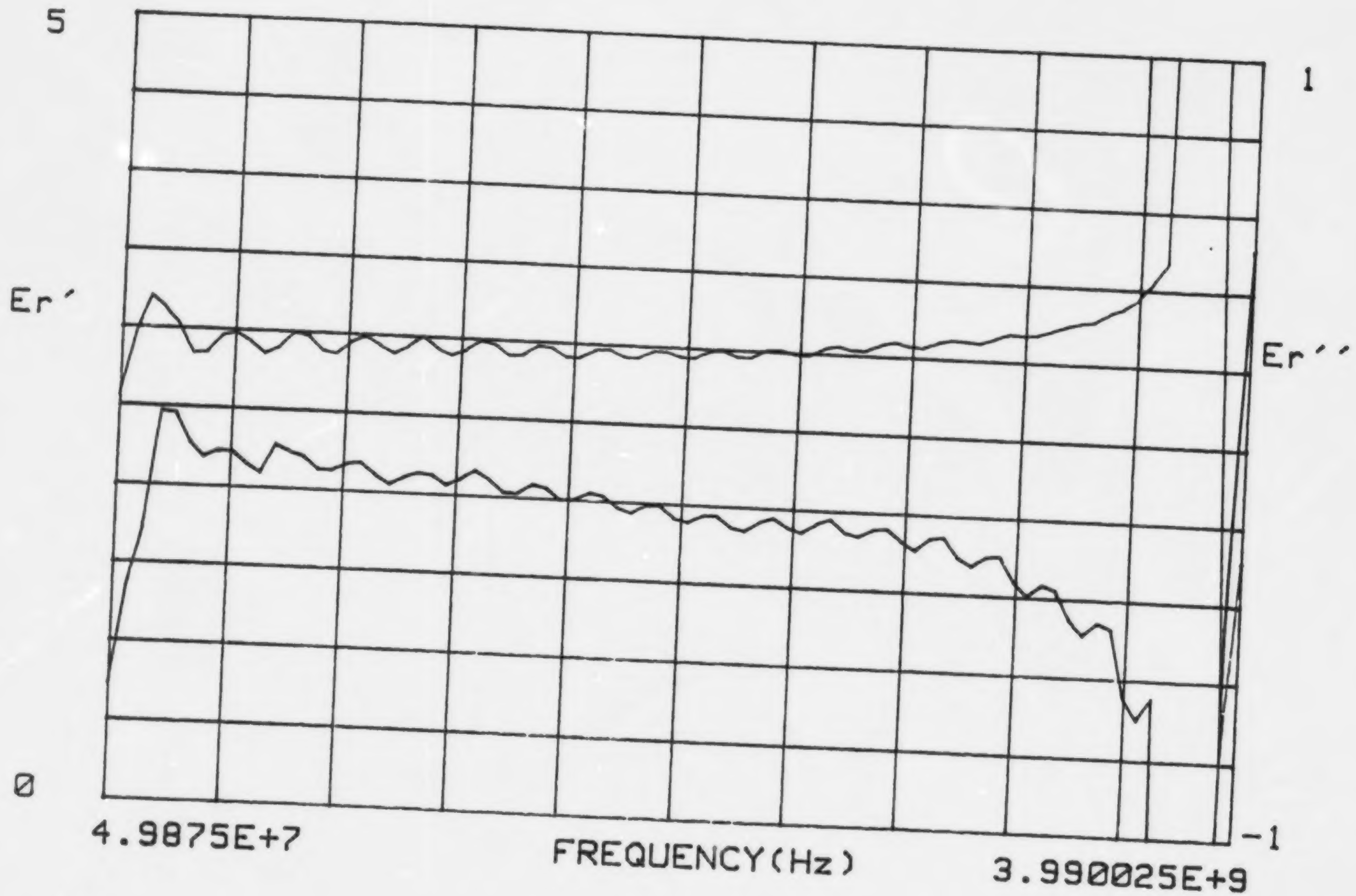


Grade A , 0.1226 % moisture

0.1301 % moisture

10:13:17 18 Jun 1988

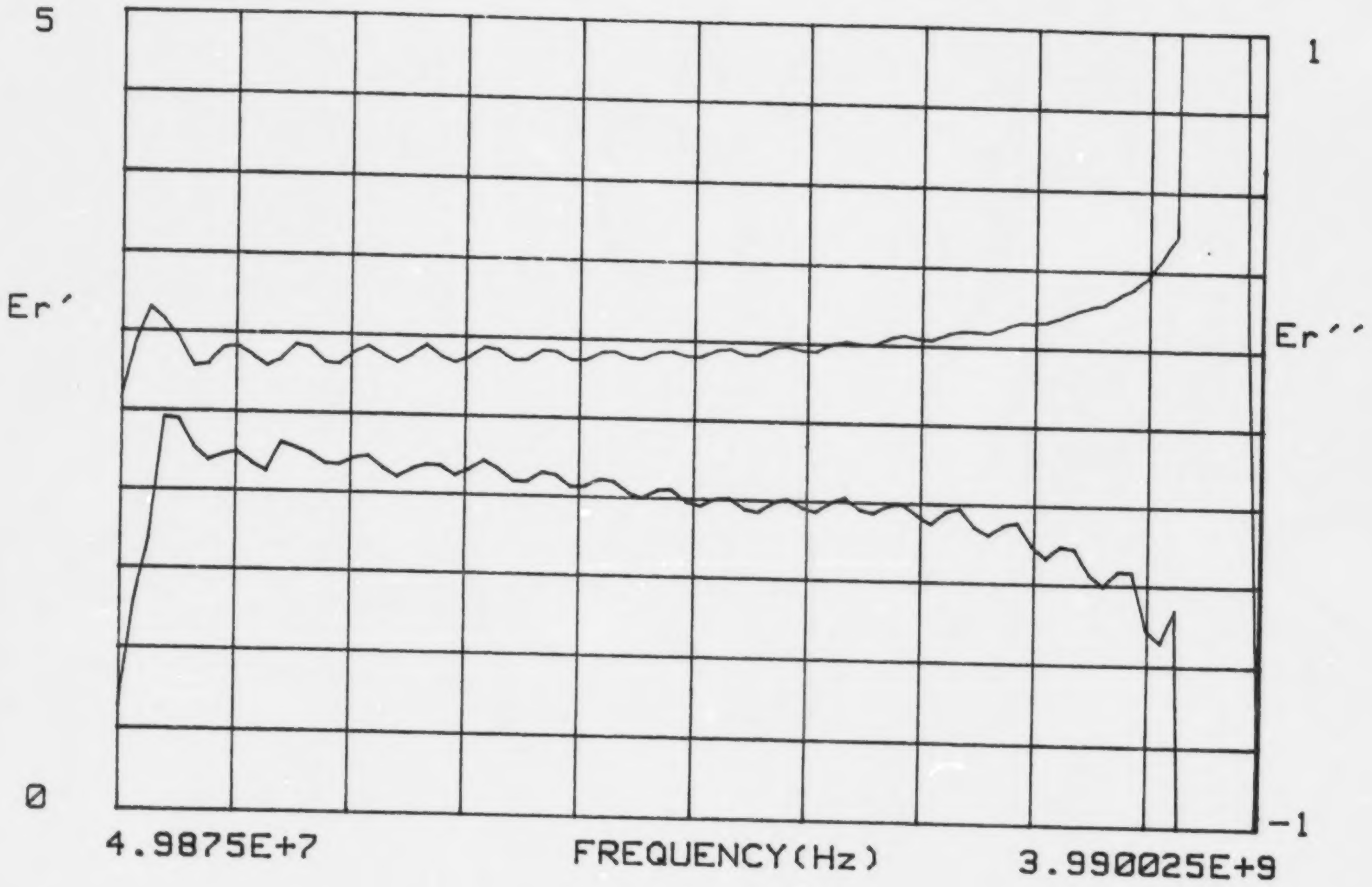
[illegible]



Grade A , 0.1301% moisture

0.1496% moisture

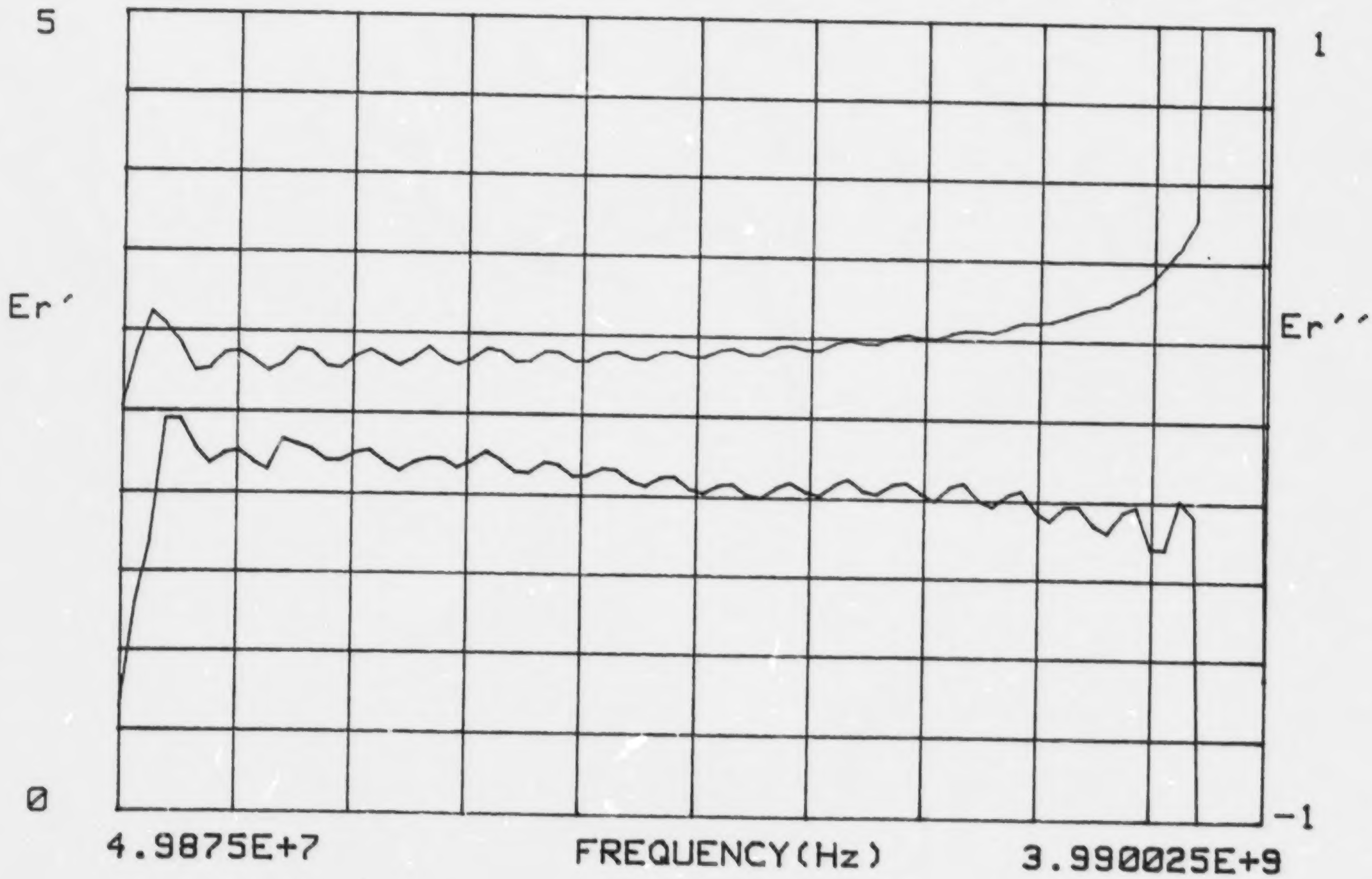
10:35:58 18 Jun 1988



Grade A , 0.1496 % moisture

0.1719% m[illegible]ture

11:03:25 18 Jun 1988

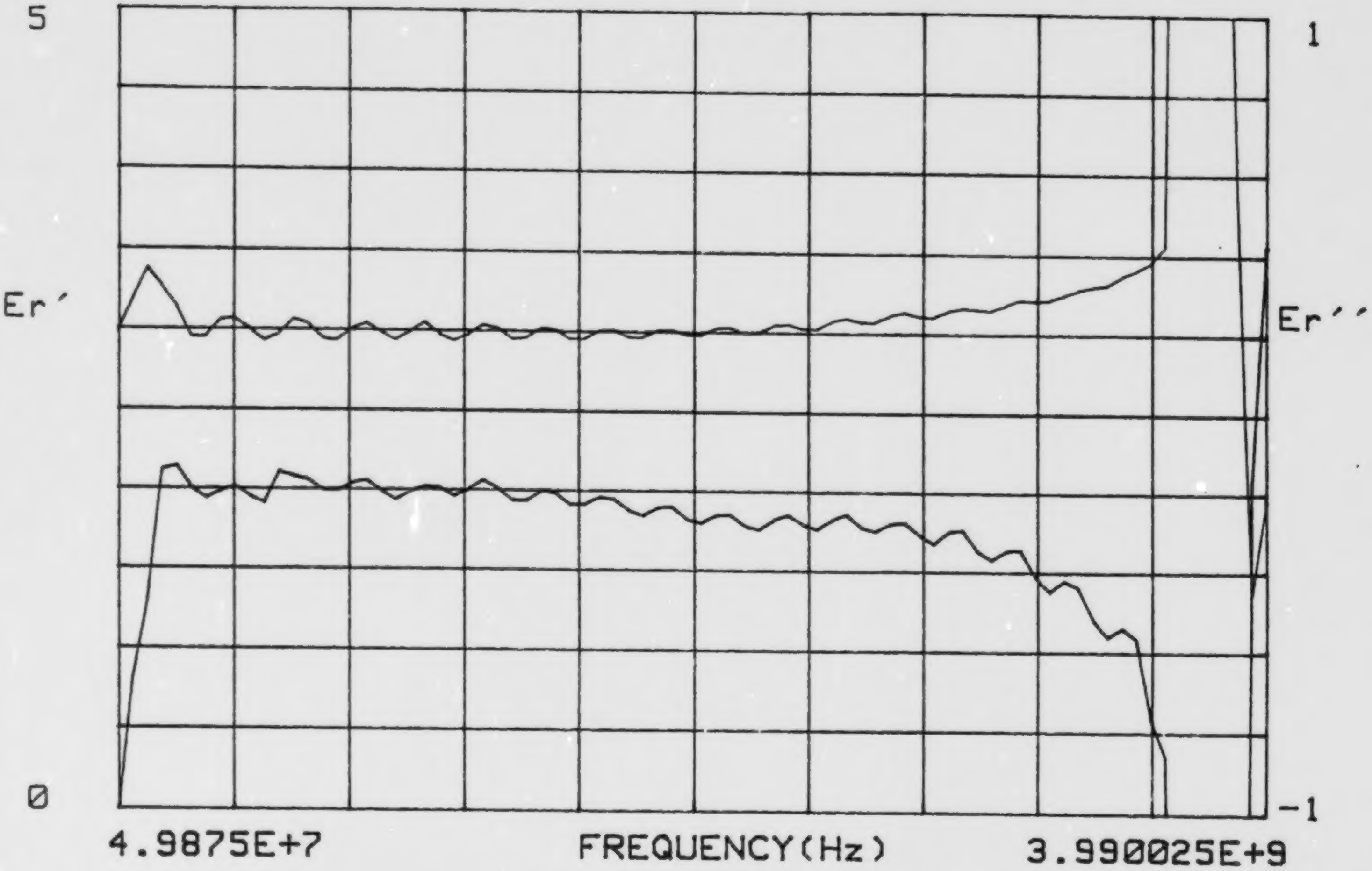


Grade A , 0.1719 moisture

0.2776% moisture

11:36:51 18 Jun 1988

5^{*} (1)

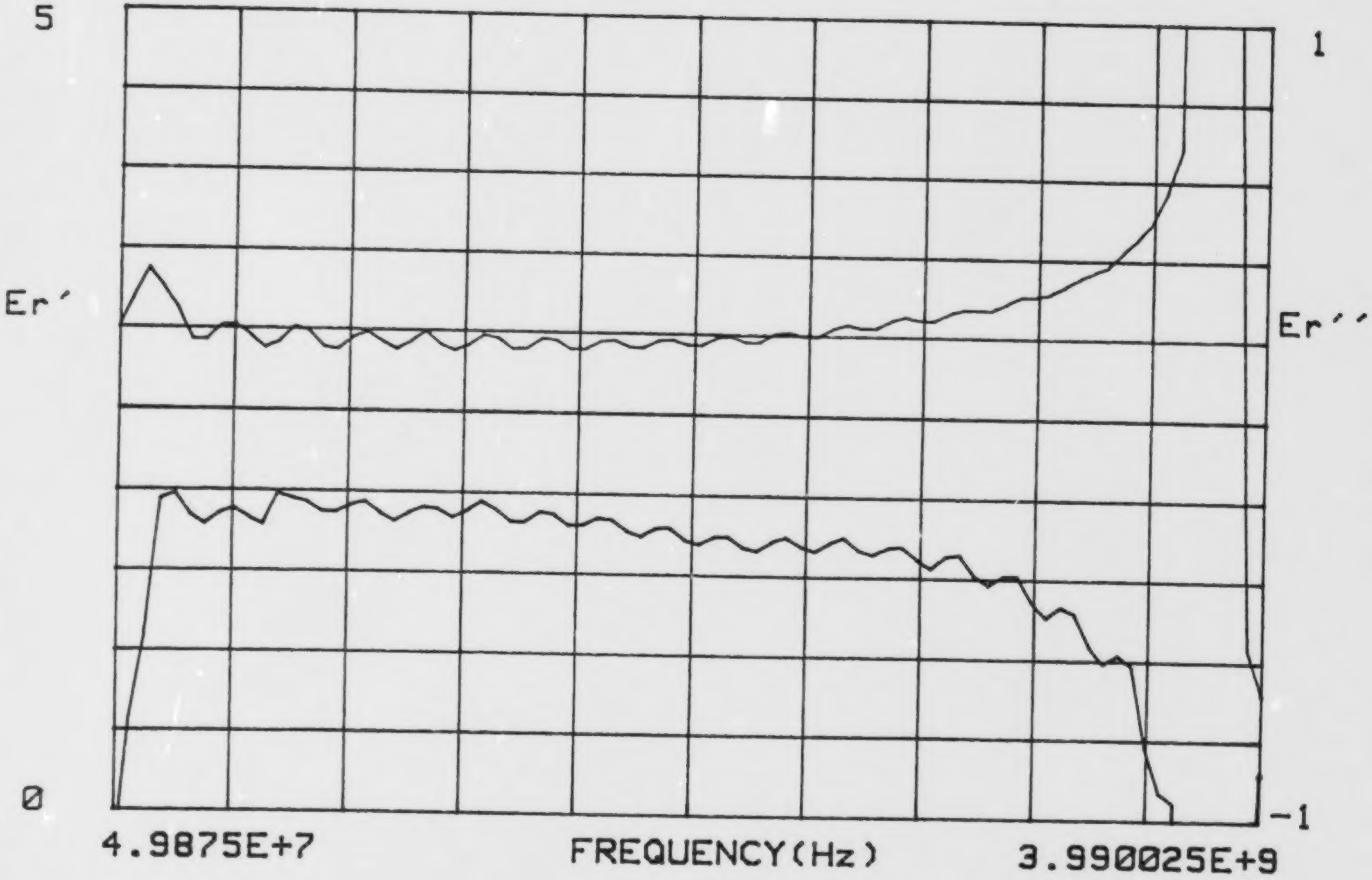


Grade A , 0.2776% moisture

0.3304% moisture

7^{r} 2)

12:08:40 18 Jun 1988

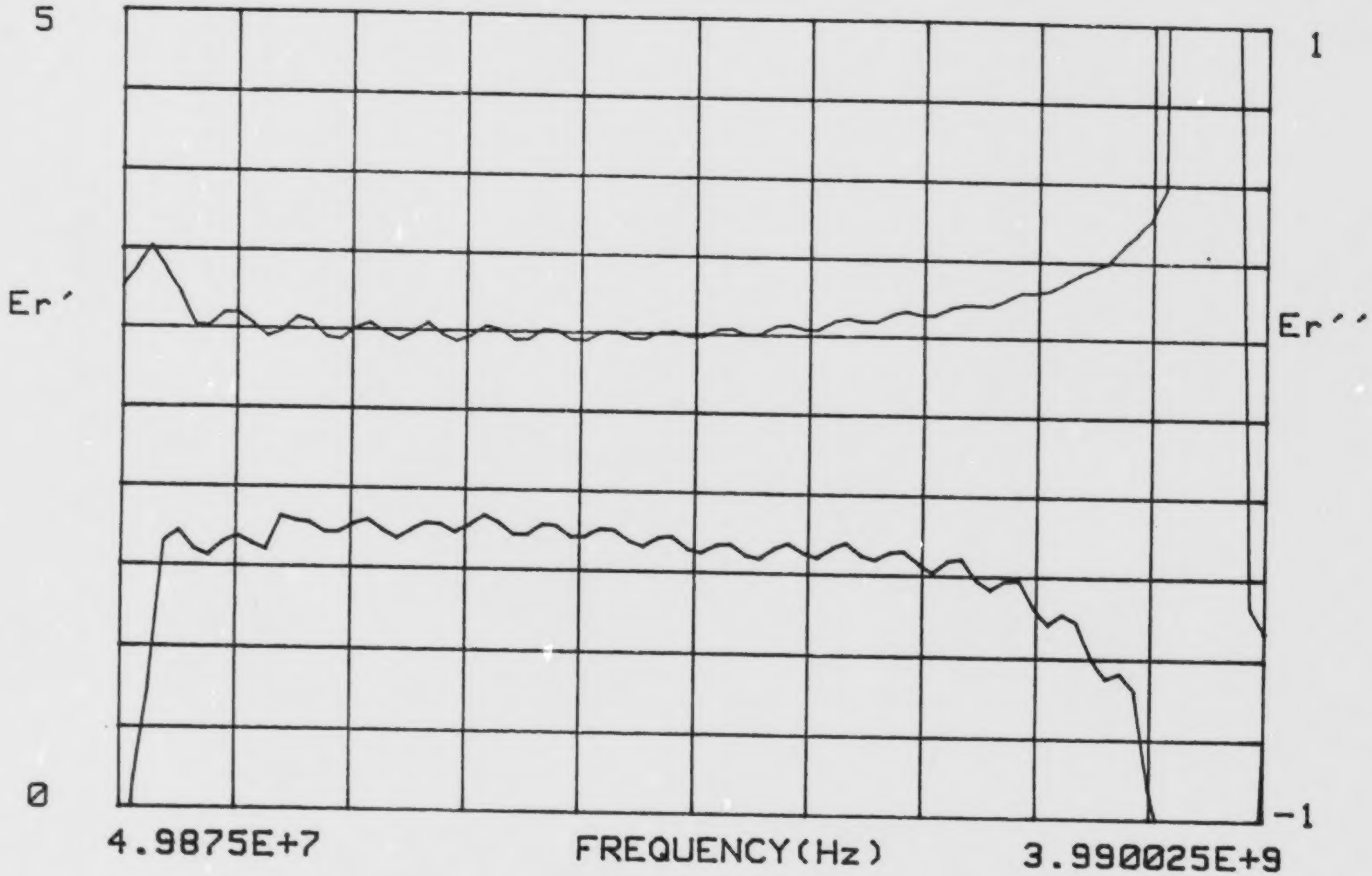


Grade A, 0.3304% moisture

0.4486% moisture

12:32:58 18 Jun 1988

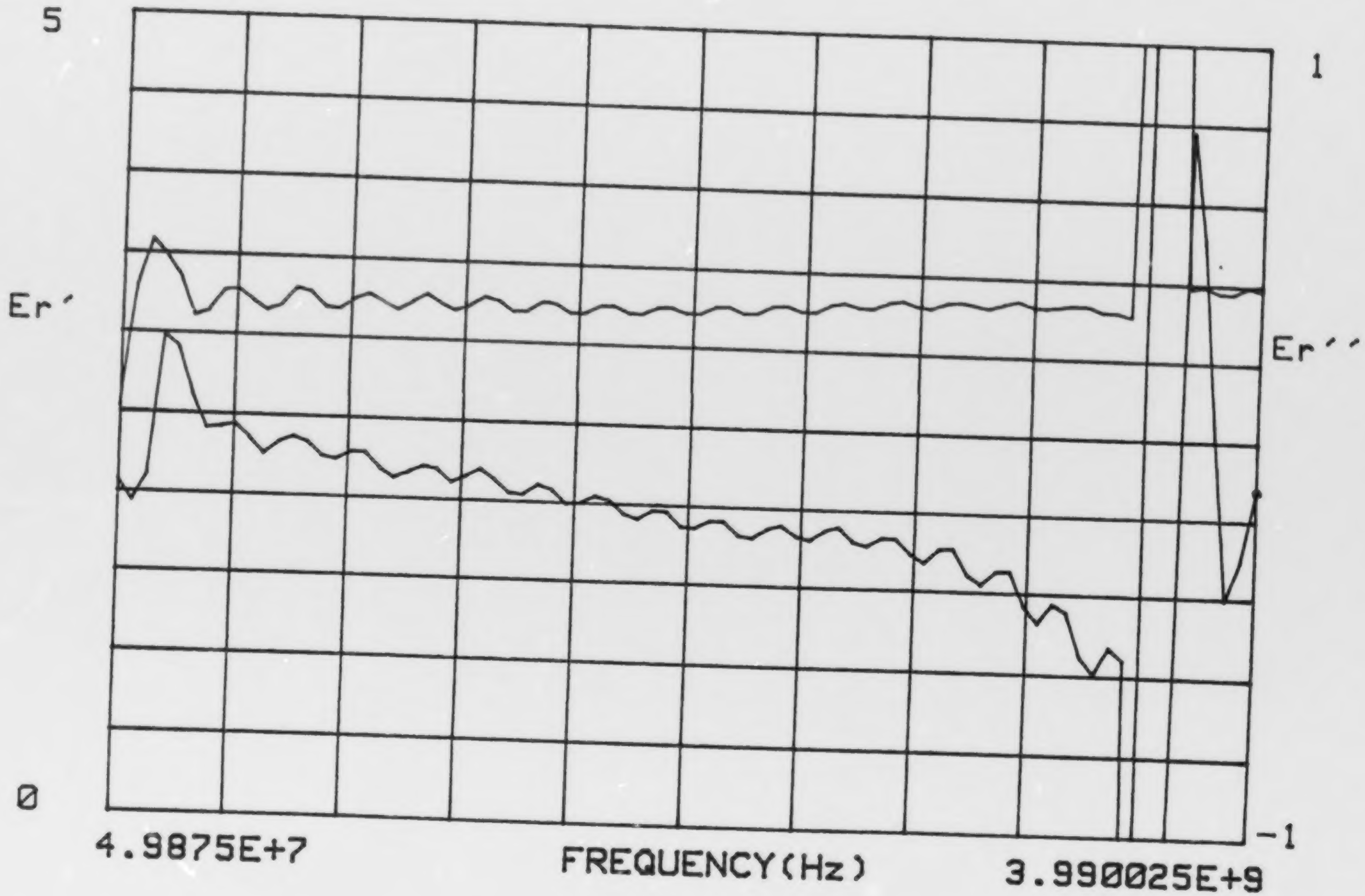
γ^{d} (3)



Grade A , 0.4486% moisture

10:05:22 19 Jun 1988

original B 0.03773% moisture

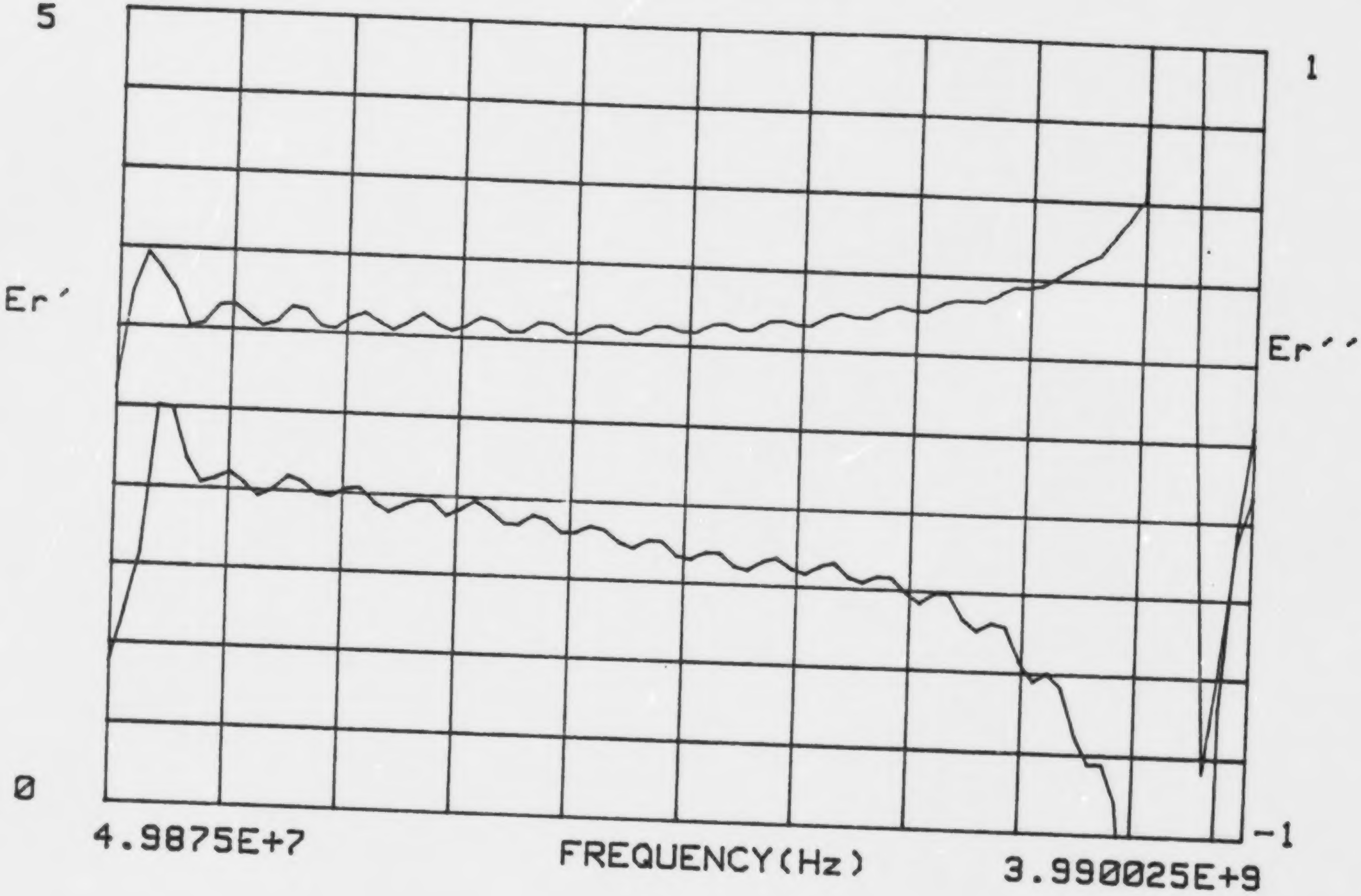


Grade B , 0.03773% moisture

10:32:11 19 Jun 1988

6

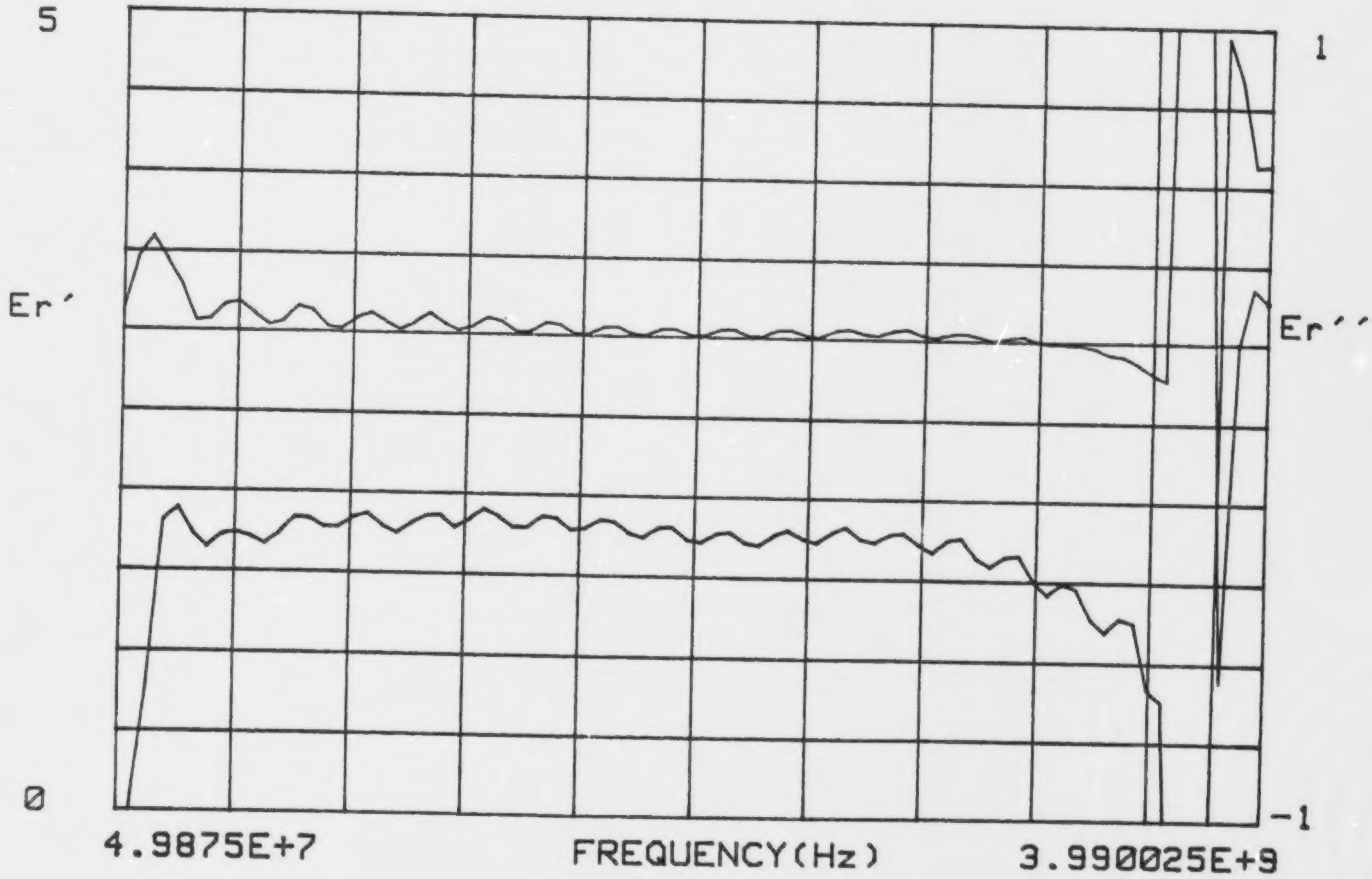
0.1127% moisture



Grade B , 0.1127% moisture

11:01:46 19 Jun 1988

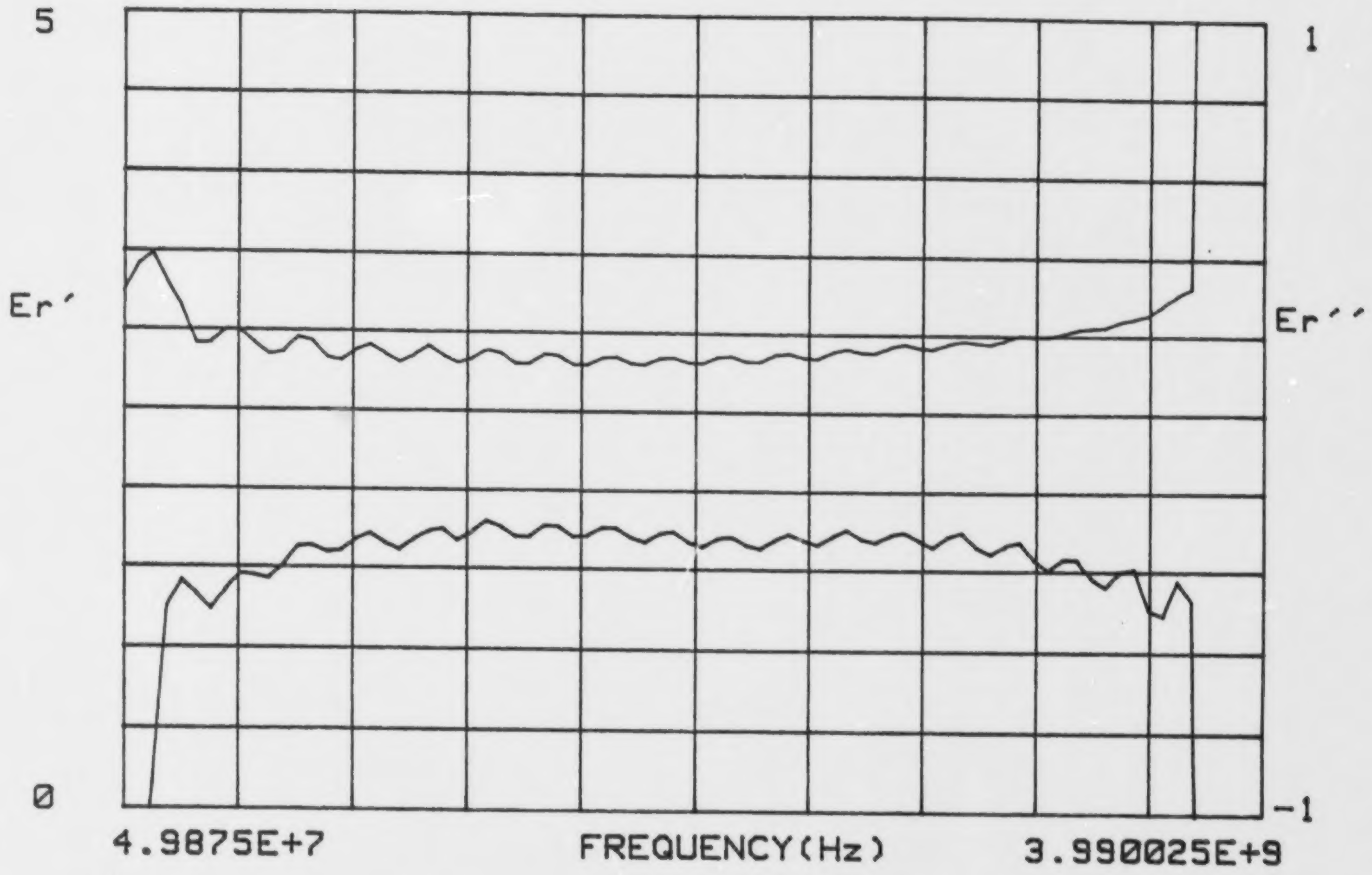
'' 0.2404% moisture



Grade B , 0.2404% moisture

11:35:20 19 Jun 1988

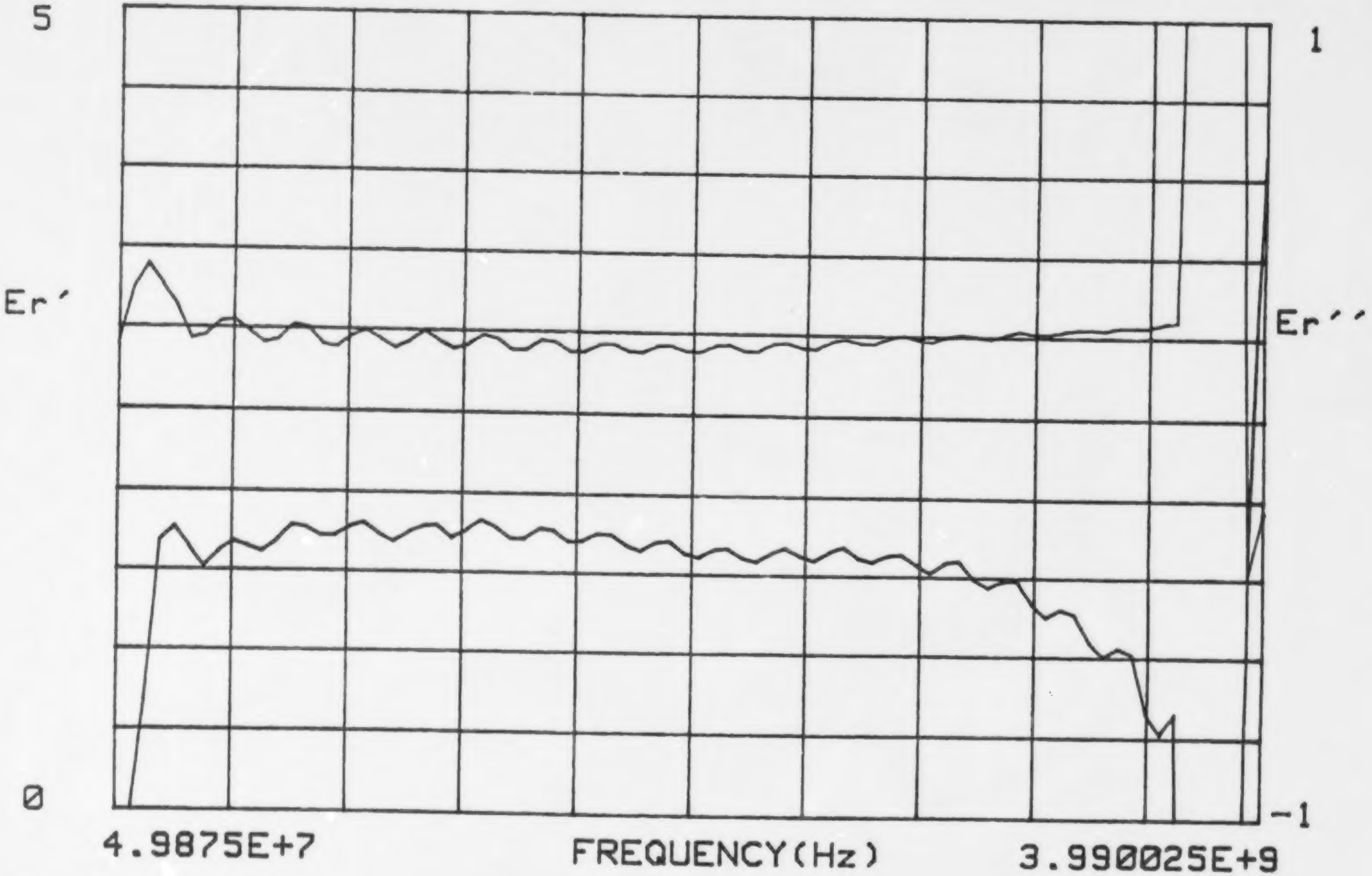
17 0.3655% moistu



Grade B , 0.3655% moisture

12:02:40 19 Jun 1988

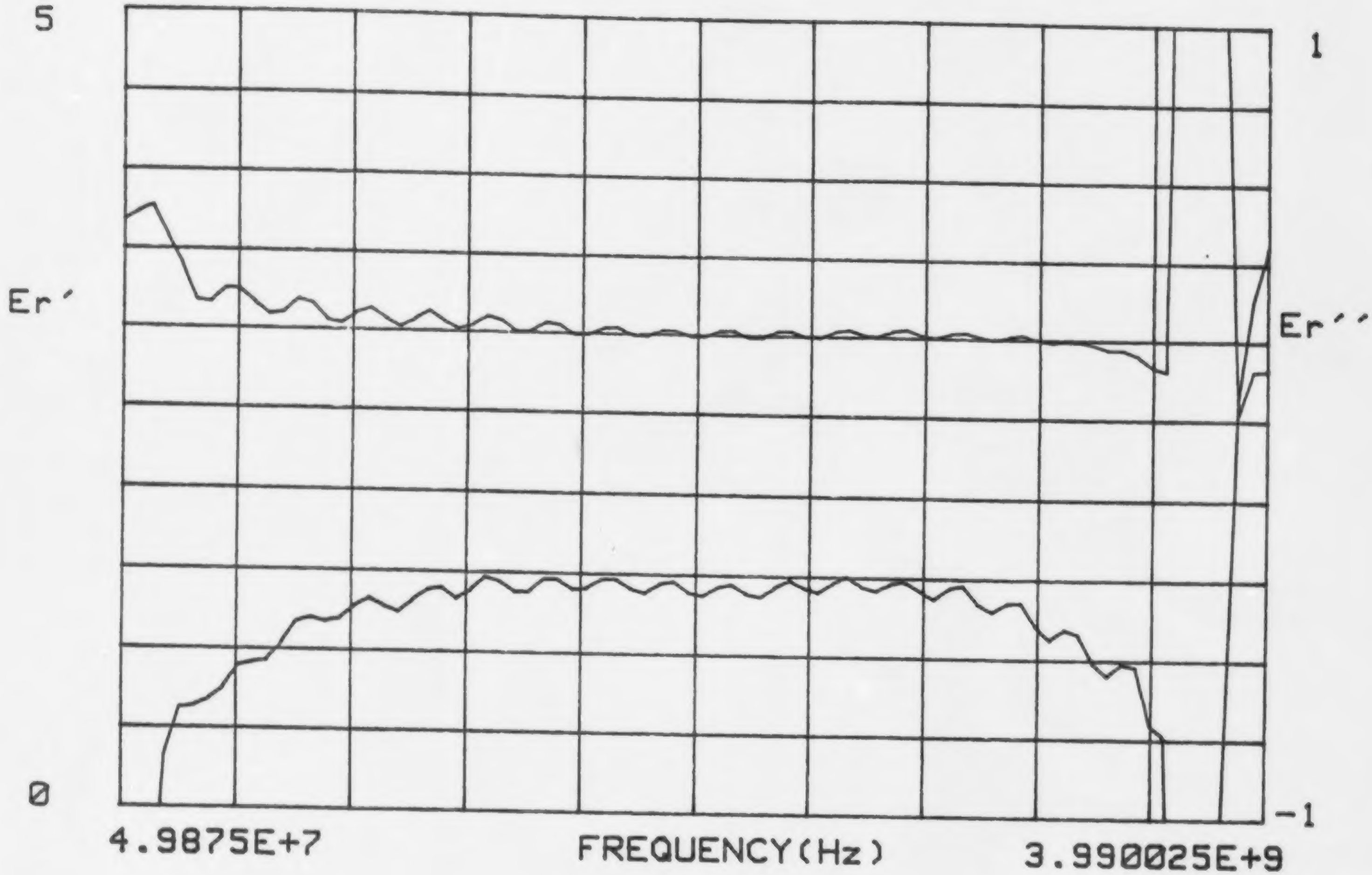
0.2971% moisture 18



Grade B , 0.2971% moisture

0.4111% moisture 5*

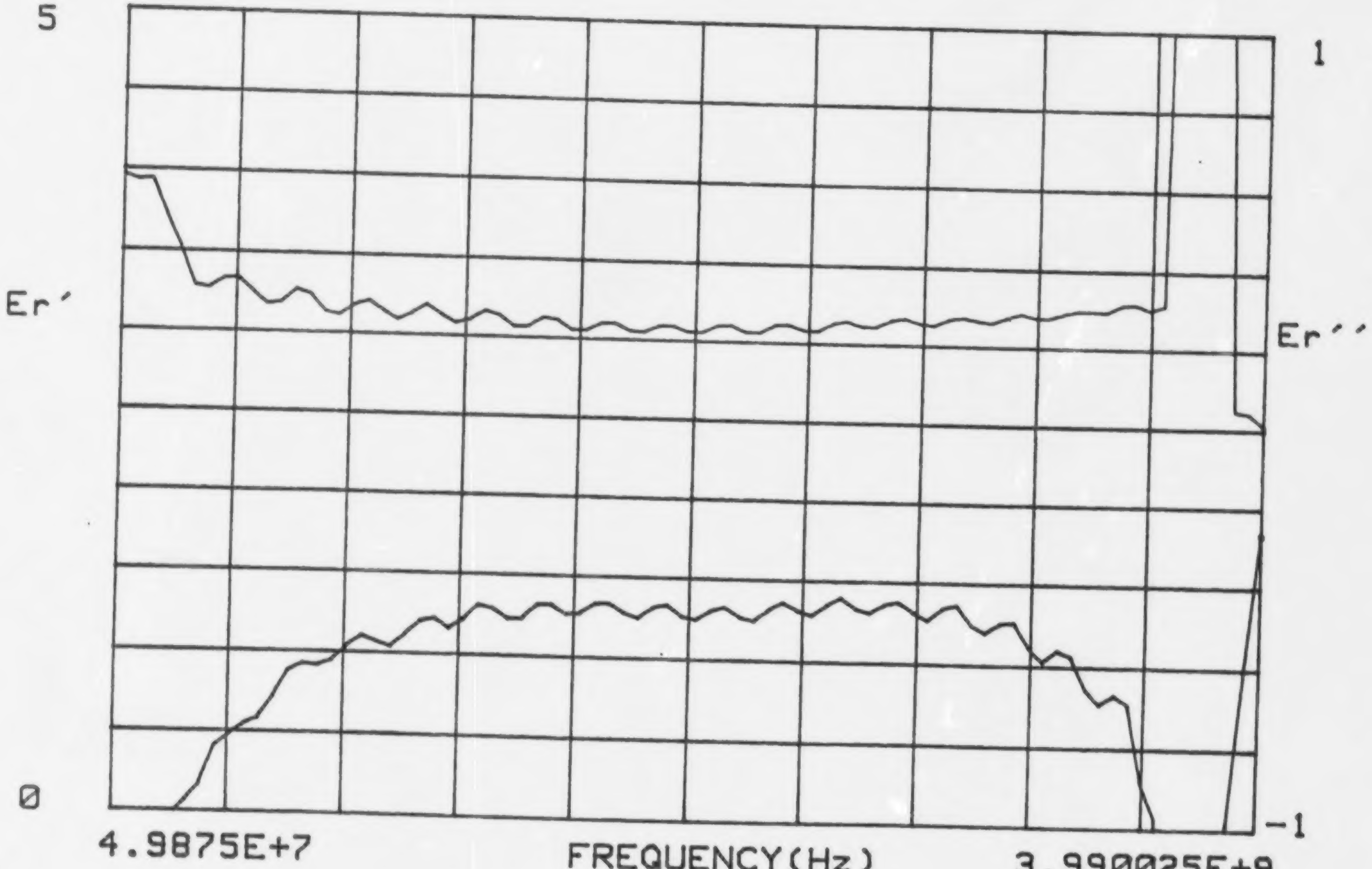
12:32:33 19 Jun 1988



Grade B , 0.4111% moisture

0.5319% moisture

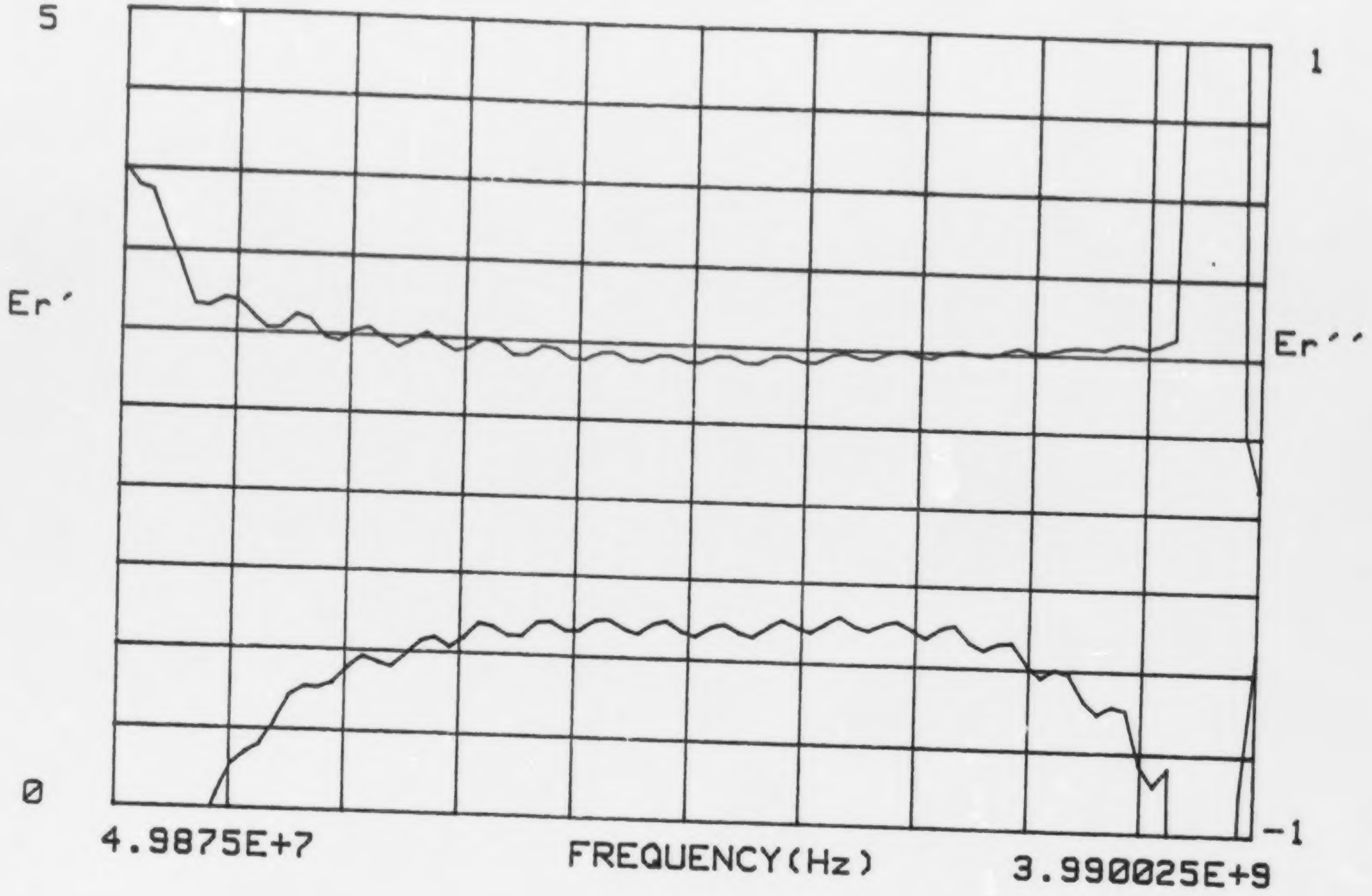
13:03:17 19 Jun 1988



Grade B , 0.5319% moisture

0.6785% moisture 8"

13:36:01 19 Jun 1988



Grade B , 0.6785 % moisture

```
10    !
20    ! Program for dielectric constant constant calculation
30    !
40    ! March 17 / 88
50    ! CENTRE FOR COMMUNICATION STUDIES
60    !
61    ! Modified March 25/88 to allow for non air dielectric design
62    !
70    ! Note:
80    !    1.  The HP 8510 network analyzer must be set up in the
90    !         following manner:
100   !             - 401 points
110   !             - channel 1  is s11
120   !             - channel 2  is s21
130   !    2.  Other recommended settings include:
131   !             - step mode
132   !             - averageing on 256
133   !             - time domain low pass mode on with gating on
134   !                  around the transmission line.
135   !             - start and stop frequencies set correctly before
136   !                  calibration for start*401=stop.  This should
137   !                  also be checked using the set low pass function,
138   !                  under the calibration menu.
140   !
141   !    3.  The plotter if connected must be attached to the computer's
142   !            system HPIB bus.  The address must be 705.
143   !
145   !    4.  The printer is assumed connected for address 701.
146   !
147   !    5.  The HP Product Note 8510-3 "Materials Measurement" was
148   !           used as a guide for this program.  Additional considerations
149   !           are as follows:
150   !                - transmission line lenght was extended
151   !                - transmission line impedance was constructed
152   !                     for a 50 ohm impedance with a dielectric
153   !                     constant of approx 3.  This should result
154   !                     in improved accuracy.
155   !
157   !
158   REAL Datatemp(1:401,1:2)   ! temp array for getting na info
160   COMPLEX S11(1:401)         ! s11 complex array from na
170   COMPLEX S21(1:401)         ! s21 complex array from na
180   REAL Strfreq               ! start freq of na
190   REAL Stpfreq               ! stop freq of na
200   ! constants necessary for calculation
210   D=.99/39.37                 ! lenght of test set in meters
220   C=3.E+8                    ! speed of light in m/sec
230   REAL W(1:401)              ! w=2*pi*freq
240   REAL L(1:401)              ! lamba (wavelenght) for all points
250   REAL Freq(1:401)           ! contains freq of every point
260   ! complex arrays necessary for calculation
270   COMPLEX K(1:401)           ! temp array
280   COMPLEX M(1:401)           ! temp array
290   COMPLEX M1(1:401)          ! temp array for m(i)
300   COMPLEX M2(1:401)          ! temp array for m(i)
310   COMPLEX T(1:401)           ! temp array
320   COMPLEX T1(1:401)          ! temp array for t(i)
330   COMPLEX U(1:401)           ! relative permeability of material
340   COMPLEX E(1:401)           ! relative permittivity of material
350   COMPLEX V(1:401)           ! temp array
360   COMPLEX V1(1:401)          ! temp array for v(i)
```

```
390   !
392   COMPLEX X(1:401)          ! temp variable x and y
393   COMPLEX Y(1:401)
394   COMPLEX Cost(1:401)
400   Prt_toggle=0              ! initialize toggle for printing 2 plots
410   !                           per page
420   Low=0                     ! low limit for plot range
430   High=5                    ! high limit for plot range
431   Low_loss=-1               ! limit for dielectric loss plot
432   High_loss=1
434   DIM Title$[50]            ! title line used for plot
440   !
450   Er=1.0                    ! estimated epsilon
460   Ur=1.                     ! estimated mew
470   !
471   White=1                   ! pen colors
472   Red=2
473   Yellow=3
474   Green=4
475   Cyan=5
476   Blue=6
477   Magenta=7
478   !
480   INTEGER Preamble,Size     ! for data from network analyzer
490   !
500   ASSIGN @Printer TO 701         ! set printer location
501   Plotter=705                    ! set plotter location
510   ASSIGN @Na TO 716              ! set HP8510 location
520   ASSIGN @Na1 TO 716;FORMAT OFF  ! set hp8510 location format off
530   PLOTTER IS CRT,"INTERNAL"      ! set crt to a plotter
540   GRAPHICS ON
550   !
560   GCLEAR
570   CLEAR @Na
580   !
590   OUTPUT @Na;"debuon;"      ! debug on
600   OUTPUT @Na;"ento;"        ! terminate previous transfers
610   !
620 Start:   !
630   CONTROL 2,2;1             ! select the user menu
640   OUTPUT 2;"scratch keyE";
650   PRINT USING "@"           ! clear the screen
660   KEY LABELS PEN 5                     ! set pen color
670   !
680 Loop:      !
690   ON KEY 1 LABEL "START          " GOSUB Start_sequence
700   ON KEY 2 LABEL "               " GOTO Loop
710   ON KEY 3 LABEL " TITLE   LINE  " GOSUB Title_line
715   ON KEY 4 LABEL " NEW    LIMITS " GOSUB New_limits
720   ON KEY 5 LABEL "PLOT TO   CRT  " GOSUB Crt_plot
740   ON KEY 6 LABEL "PLOT TO PLOTTER" GOSUB Plotter_dump
750   ON KEY 7 LABEL "PLOT TO PRINTER" GOSUB Printer_dump
760   ON KEY 8 LABEL " LIST   RANGE  " GOTO List_range
770   GOTO Loop
780   !
790   !
791 Start_sequence:    !
792   Title$=" "          ! initialize title line
794   GOSUB Get_s11_s21
795   GOSUB Calculate
796   GOSUB Crt_plot
797   RETURN
798   !
799   !
```

```
820   ENTER @Na1;Preamble,Size,Datatemp(*)
830   ! next convert to complex array
840   FOR I=1 TO 401
850     S11(I)=CMPLX(Datatemp(I,1),Datatemp(I,2))
860   NEXT I
870   !
880   OUTPUT @Na;"chan2;form3;outpdata;";  ! get ch2 data (complex)
890   ENTER @Na1;Preamble,Size,Datatemp(*)
900   ! next convert to complex array
910   FOR I=1 TO 401
920     S21(I)=CMPLX(Datatemp(I,1),Datatemp(I,2))
930   NEXT I
940   !
950   ! get the frequency information
960   OUTPUT @Na;"star;outpacti;"     ! select start freq then active function
970   ENTER @Na;Strfreq
980   OUTPUT @Na;"stop;outpacti;"     ! get stop freq
990   ENTER @Na;Stpfreq
1000  RETURN
1010  !
1020  !
1100  !
1110  !
1120 Calculate:   !
1130  !
1140  ! this subroutine calculates epsilon r and mew r
1150  !    given s11(i) [complex]
1160  !          s21(i) [complex]
1170  !          d  lenght of test set in meters
1180  !          c  speed of light in m/sec
1190  !
1200  ! first compute the freq array freq(i) for the 401 points
1210  Inc=(Stpfreq-Strfreq)/400
1220  Freq(1)=Strfreq
1230  FOR I=2 TO 401
1240    Freq(I)=Freq(I-1)+Inc
1250  NEXT I
1260  !
1270  ! next step calculate the W(i) array. Where w=2*pi*freq
1280  !  note this is an array of 401 frequencies
1290  !  also lamba [l(i)] can be calculated at the same time
1300  FOR I=1 TO 401
1310    W(I)=2*PI*Freq(I)
1320    L(I)=C/Freq(I)
1330  NEXT I
1340  !
1341  Ncwn=0          ! Initialize counter
1350  ! calculate paramcters
1360  FOR I=1 TO 400
1361  ! S11(I)=CMPLX(-.260970154,.676652054)*S11(I)
1362  ! S21(I)=CMPLX(-3.557319103,1.371985097)*S21(I)
1363   !S11(I)=CMPLX(.0066256,-.0746207)
1364  !S21(I)=CMPLX(-.99330691,.08815905)
1365  ! S21(I)=S21(I+1)
1366  !IF ABS(REAL(S11(I)))<.04 THEN
1367  !M1(I)=COS(4*PI*Freq(I)*D/C)
1368  !ELSE
1370    K(I)=((S11(I)^2-S21(I)^2)+1)/(2*S11(I))
1380    M1(I)=K(I)+SQRT(K(I)^2-1)
1390    M2(I)=K(I)-SQRT(K(I)^2-1)
1391  ! END IF
1400    ! check which +,- is the correct value
1410    IF ABS(M1(I))<=1 THEN
1420       M(I)=M1(I)
```

```
1450     END IF
1451    !IF ABS(REAL(S11(I)))<.04 THEN
1452   ! T(I)=S21(I)
1453   ! ELSE
1460     T(I)=((S11(I)+S21(I))-M(I))/(1-(S11(I)+S21(I))*M(I))
1461   ! END IF
1470     ! calculate the n value
1492    Cost(I)=CMPLX(COS(4.*PI*D*Freq(I)/C),-SIN(4.*PI*D*Freq(I)/C))
1493    IF I>1 THEN
1495   IF ARG(S21(I)*(1.-.3054*Cost(I)))>ARG(S21(I-1)*(1-.3054*Cost(I-1)))+PI/4 T
1496    ! IF ARG(S21(I))>ARG(S21(I-1))+(1/2)*PI THEN
1497        Neun=Neun+1
1498        END IF
1499    END IF
1501    N(I)=Neun
1504     !
1505 ! N(I)=INT(D*(Freq(I)*SQRT(Er*Ur))/C)
1506  ! N(I)=1
1508    !  N(I)=INT(I/25)
1509    !FOR J=1 TO 9
1510    !N(J)=0
1511    !NEXT J
1512    !FOR J=10 TO 29
1513    !N(J)=1
1514   ! NEXT J
1515    !FOR J=30 TO 49
1516    !N(J)=2
1517    !NEXT J
1518    !FOR J=50 TO 58
1519   ! N(J)=3
1520   ! NEXT J
1521 !  FOR J=59 TO 401
1522   ! N(J)=INT(D*Freq(J)/C/SQRT(Ur*Er))
1523   ! NEXT J
1524     !
1525     ! create the modified T1(i) array based on n
1526     T1(I)=LOG(1/T(I))
1527     T1(I)=CMPLX(REAL(T1(I)),IMAG(T1(I))+2*PI*N(I))
1528     V2(I)=-(1/(2*PI*D)*T1(I))^2
1529     V(I)=SQRT(V2(I))
1530     IF REAL(V(I))>=0 THEN
1531     V(I)=V(I)
1532     ELSE
1533     V(I)=-V(I)
1534     END IF
1535     !
1536     V1(I)=1/V(I)
1537     U(I)=50.*(1+M(I))/(V1(I)*173.553*(1-M(I)))*L(I)
1540     E(I)=(1/(V1(I)^2))*(L(I)^2)/U(I)
1601     ! Y(I)=-(C/(2*PI*Freq(I)*D)*T1(I))^2
1602     ! X(I)=(50*(1+M(I))/(88.9*(1-M(I))))^2
1603     !
1610     ! U(I)=SQRT(X(I)*Y(I))
1620     ! E(I)=SQRT(Y(I)/X(I))
1646       !
1647  NEXT I
1648  RETURN
1650  !
1660  !
1661 Title_line:   !
1662  ! This subroutine gets a user defined title line for the plot
1663  DISP "Enter the title line info";
1664  INPUT Title$
1665  GOSUB Crt_plot
```

```
1669  !
1670 Draw_all:    !
1680  !
1690  PRINT USING "@"           ! clear the alpha screen
1700  GCLEAR                    ! clear the graphics screen
1780  ! first label the axis
1790  PEN White                    ! set color to white
1791  ! set up a viewport at top of screen
1793  VIEWPORT 0,120,0,100
1794  WINDOW 0,120,0,100
1795  CSIZE 3.5
1796  MOVE 10,95
1800     LABEL TIME$(TIMEDATE);"  ";DATE$(TIMEDATE)
1801     LABEL Title$
1810  MOVE 0,87                    ! label left top
1811    PEN Green
1820    LABEL High
1830  MOVE 0,19                    ! label left bottom
1831    LABEL Low
1833  MOVE 111,87                  ! label right top
1834    PEN Blue
1836    LABEL High_loss
1837  MOVE 111,19                  ! label right bottom
1838    LABEL Low_loss
1839  !   MOVE 0,52                    ! label Ur (left)
1840  !     PEN Red
1841  !     LABEL "Ur"
1842  !   MOVE 111,52                  ! label Ur loss right side
1843  !     PEN Cyan
1844  !     LABEL "Ur"
1845  !     LABEL " loss"
1846  MOVE 0,62                    ! label Er left
1847    PEN Green
1848    LABEL "Er'"
1849  MOVE 111,62                  ! label Er loss (right)
1850    PEN Blue
1851    LABEL "Er''"
1853  PEN Yellow
1854  MOVE 7,15
1855    LABEL PROUND(Strfreq,3)
1856  MOVE 50,15
1857    LABEL "FREQUENCY(Hz)"
1858  MOVE 87,15
1859   ! LABEL PROUND(Stpfreq,3)
1860   LABEL PROUND(Freq(80),3)
1861  !
1862  ! draw the axis
1863  PEN Yellow                   ! set axis color to yellow
1864  VIEWPORT 10,110,20,90
1865  WINDOW 0,20,0,20
1866  GRID 2,2,10,10
1867  !
1868  GOSUB Ur_plot
1869  GOSUB Er_plot
1870  !
1871  RETURN
1872  !
1873  !
1874 New_limits:   !
1875  DISP "Enter the new lower and upper limits [low,high]";
1876  INPUT Low,High
1877  GOSUB Crt_plot
1878  RETURN
1879  !
---
```

```
1882                RETURN
1883  ! draw U(i) mew in red
1884  PEN Red
1885  WINDOW 1,401,Low,High
1886  MOVE 1,REAL(U(1))
1887  FOR I=2 TO 401
1888    DRAW I,REAL(U(I))
1889  NEXT I
1890  ! draw U(i) mew imaginary part (loss) in cyan
1891  PEN Cyan
1892  WINDOW 1,401,Low_loss,High_loss
1893  MOVE 1,IMAG(U(1))
1894  FOR I=2 TO 4
1895    DRAW I,IMAG(U(I))
1896  NEXT I
1897  RETURN
1898  !
1899  !
1900 Er_plot:    !
1901  ! draw E(i) eplison in green
1902  PEN Green
1903  WINDOW 1,80,Low,High
1904  MOVE 1,REAL(E(1))
1905  FOR I=2 TO 401
1906    DRAW I,REAL(E(I))
1907  NEXT I
1908  ! draw E(i) eplison  loss imginary in blue
1909  PEN Blue
1910  WINDOW 1,80,Low_loss,High_loss
1911  MOVE 1,IMAG(E(1))
1912  FOR I=2 TO 401
1913    DRAW I,IMAG(E(I))
1914  NEXT I
1915  RETURN
1916  !
1917  !
1918  !
1919  !
1920 Crt_plot:   !
1921  PLOTTER IS CRT,"INTERNAL"
1922  GOSUB Draw_all
1923  RETURN
1924  !
1925 Plotter_dump:   !
1926  PLOTTER IS Plotter,"HPGL"
1927  GOSUB Draw_all
1928  PLOTTER IS CRT,"INTERNAL"
1929  RETURN
1930  !
1931 Printer_dump:   !
1932  OUTPUT 2;"!";      ! turn menu off
1933  DUMP GRAPHICS
1934  IF Prt_toggle=1 THEN
1935     OUTPUT @Printer;"
```

```
                                   ";
1936      Prt_toggle=0
1937    ELSE
1938      Prt_toggle=1
1939   END IF
1940   OUTPUT 2;"!";        ! turn menu on again
1941   RETURN
1942   !
1943   !
1944 List_range:    !
1945   DISP "Enter the # to be listed (1 to 401)";
1946   INPUT I
1947         ! printing routine
1948         GCLEAR
1949         PRINT USING "@"
1950         PRINT "FREQ = ";Freq(I)
1951         PRINT "LAMBDA = ";L(I)
1952         PRINT TABXY(0,0);"i =";I
1953         PRINT "S11=";S11(I)
1954         PRINT "s21=";S21(I)
1955         PRINT "K = ";K(I)
1956         PRINT "M = ";M(I)
1957         PRINT "     where M1 = ";M1(I)
1958         PRINT "           M2 = ";M2(I)
1959         PRINT "N value =";N(I)
1960         PRINT "T = ";T(I)
1961         PRINT "     where t1 = ";T1(I)
1962         PRINT "V = ";V(I)
1963         PRINT "X = ";X(I)
1964         PRINT "Y = ";Y(I)
1965         PRINT "E = ";E(I)
1966         PRINT "U = ";U(I)
1967    GOTO List_range
1968   END
```

APPENDIX II

THE MEASUREMENT OF THE MOISTURE CONTENT IN PROCESSED POTASH USING MICROWAVE TECHNIQUES

(Supplementary Report)

UNIVERSITY OF SASKATCHEWAN

DEPARTMENT OF
ELECTRICAL ENGINEERING

SASKATOON, CANADA
S7N 0W0

August 24, 1988

Dan Gauthier, P.Eng.
Process Engineer
Suite 500, 122-1st Ave. S.
Saskatoon, Sask.
S7K 6A3

Dear Dan:

A supplementary report (dated July 27, 1988) which summarizes the results of the work done under the project "Measurement of the Moisture Content in Processed Potash Using Microwave Techniques" is attached. This report also indicates the possible future work in this area. If you have any questions please do not hesitate to contact me.

Yours sincerely,

Surinder Kumar
Professor of
Electrical Engineering

SK/ib
Attach.

SUPPLEMENTARY REPORT

Table II in the original report gives imaginary part of the dielectric constant as a function of moisture constant at three frequencies and for two grades of potash. There was a scaling factor of X5 in this table. This factor has been removed and a modified table is attached. The results from this table are also graphically shown in two figures attached with this report.

From these figures a clear trend indicating increase in ε'' with moisture constant is evident. Frequencies in the 400-800 MHz range appear to be more suitable for moisture measurement as the percentage change in ε'' is more. The sensitivity of ε'' to change in percentage moisture is more for finer grain potash than granular potash.

A discontricity in curves at all frequencies for moisture constant in the range of .1 to .2 percent was observed for granular potash. This appears to be a measurement error.

Any instrument which is designed to measure moisture constant through measurement of ε'' will also have to take into account the effect of bulk density as this is a factor in the relationship between ε'' and percentage moisture.

Measurement of a conductivity (by measuring DC resistance) were also taken for two grades of potash.

$\sigma = .2724 \times 10^{-6}\ \text{ohm}^{-1}\ \text{cm}^{-1}$ for finer grade and

$\sigma = .1872 \times 10^{-6}\ \text{ohm}^{-1}\ \text{cm}^{-1}$ for granular potash.

From this effective loss factor at 1.5 GHz was calculated at 2 GHz for .24 percent moisture for lanigan potash. This value is given by

$$\Sigma\varepsilon'' = \varepsilon'' + \frac{\sigma}{2\pi f \varepsilon_0}$$

$$= .32 + 1.685 \times 10^{-3} \approx .3217$$

For granular potash at 2 GHz and with a moisture content of .1496%

$$\Sigma\varepsilon'' = .2425$$

As materials with $\Sigma\varepsilon''$ between 2 and 100 are suitable for microwave drying it may be concluded that microwave drying is not a suitable method for this low moisture level.

However, as ε'' approaches 2 with moisture constant increase to 1 percent or so this may become feasible.

Future Direction

There are two areas for future work.

(a) The measured data was not enough to establish an empirical formula which relates ε" to moisture constant for a given frequency and the bulk density. One area of work could be measurement of more data to establish this relationship. Standard curve fitting methods are available to establish this relationship once sufficient data is measured.

(b) Another area of future work would be to explore the feasibility of a 'radar' type instrument to measure remotely the moisture constant in potash pile. Measured results indicate a clear correlation between moisture content and ε" and indicate possibility of such an instrument.

Freq \ Moisture	0.03588%	0.1226%	0.1301%	0.1496%	0.1719%	0.2780%	0.3304%	0.4486%
400 Mhz	0.04	0.08	0.12	0.12	0.10	0.20	0.25	0.33
800 Mhz	0.07	0.12	0.13	0.12	0.11	0.19	0.23	0.3
2000 Mhz	0.18	0.23	0.24	0.22	0.19	0.28	0.33	0.35

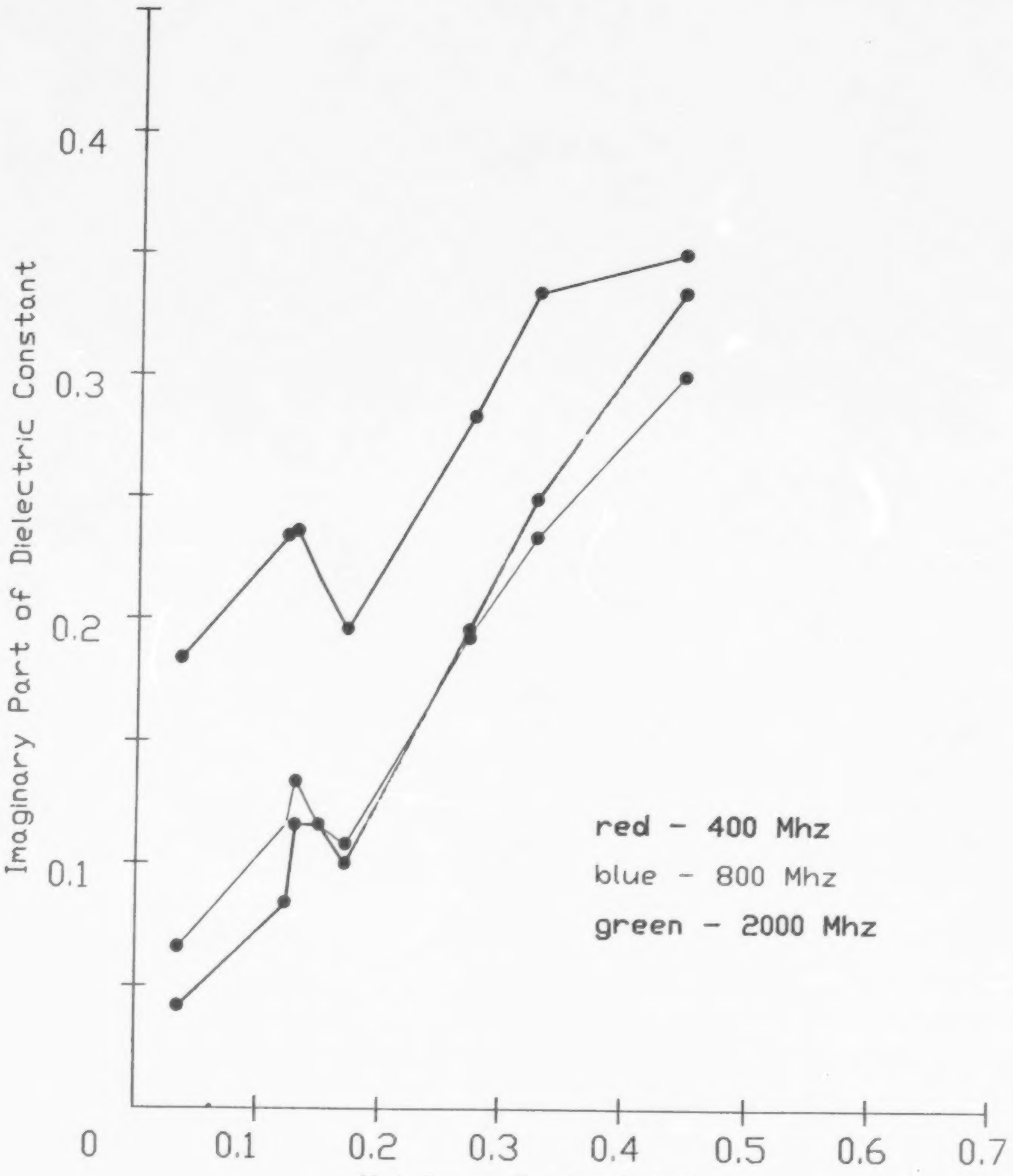
TABLE 1

The Dielectric Loss Measurements for Lanigan Granular
(no treatment)

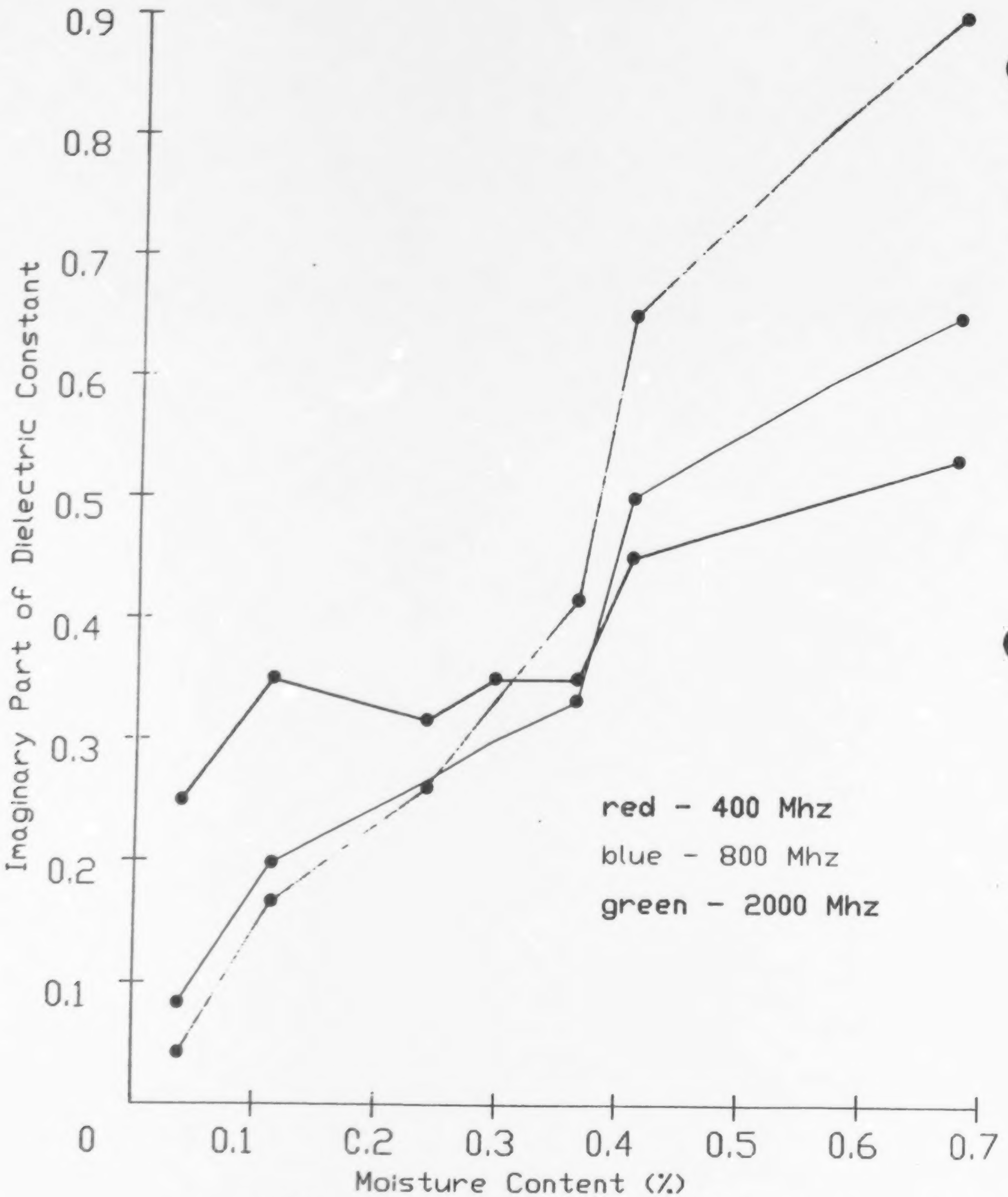
Freq \ Moisture	0.0377%	0.113%	0.240%	0.297%	0.366%	0.411%	0.572%	0.679%
400 Mhz	0.04	0.17	0.26	0.33	0.42	0.65	0.81	0.90
800 Mhz	0.08	0.20	0.27	0.30	0.33	0.50	0.60	0.65
2000 Mhz	0.25	0.35	0.32	0.35	0.35	0.45	0.50	0.53

TABLE 2

The Dielectric Loss Measurements for Lanigan Standard
(finer particle size - no treatment)



Dielectric Loss for Lanigan Granular



Dielectric Loss for Lanigan Standard

APPENDIX III

LINEAR REGRESSION ANLAYSIS

REGRESSION ANALYSIS

Least Squares Method

y = % Moisture in product (surface moisture)
x = Dielectric loss of product
r = Coefficient of correlation
r^2 = Coefficient of determination
σ = Standard deviation of "y" for a given "x"

GRANULAR PRODUCT

a) 400 Mhz "y" = 0.0079 + 1.3148 "x"
with $r = 0.9887$
$r^2 = 0.9776$
$\sigma = 0.022$

b) 800 Mhz "y" = −0.0678 + 1.7397 "x"
with $r = 0.9834$
$r^2 = 0.9670$
$\sigma = 0.026$

c) 2000 Mhz "y" = −0.2975 + 2.0035 "x"
with $r = 0.9320$
$r^2 = 0.8687$
$\sigma = 0.052$

STANDARD PRODUCT

a) 400 Mhz "y" = 0.0314 + 0.688 "x"
with $r = 0.9819$
$r^2 = 0.9641$
$\sigma = 0.045$

b) 800 Mhz "y" = −0.0507 + 1.065 "x"
with $r = 0.9782$
$r^2 = 0.9569$
$\sigma = 0.049$

c) 2000 Mhz "y" = −0.4788 + 2.1117 "x"
with $r = 0.9350$
$r^2 = 0.8743$
$\sigma = 0.083$

Moisture Freq	0.03588%	0.1226%	0.1301%	0.1496%	0.1719%	0.2780%	0.3304%	0.4486%
400 Mhz	0.04	0.08	0.12	0.12	0.10	0.20	0.25	0.33
800 Mhz	0.07	0.12	0.13	0.12	0.11	0.19	0.23	0.3
2000 Mhz	0.18	0.23	0.24	0.22	0.19	0.28	0.33	0.35

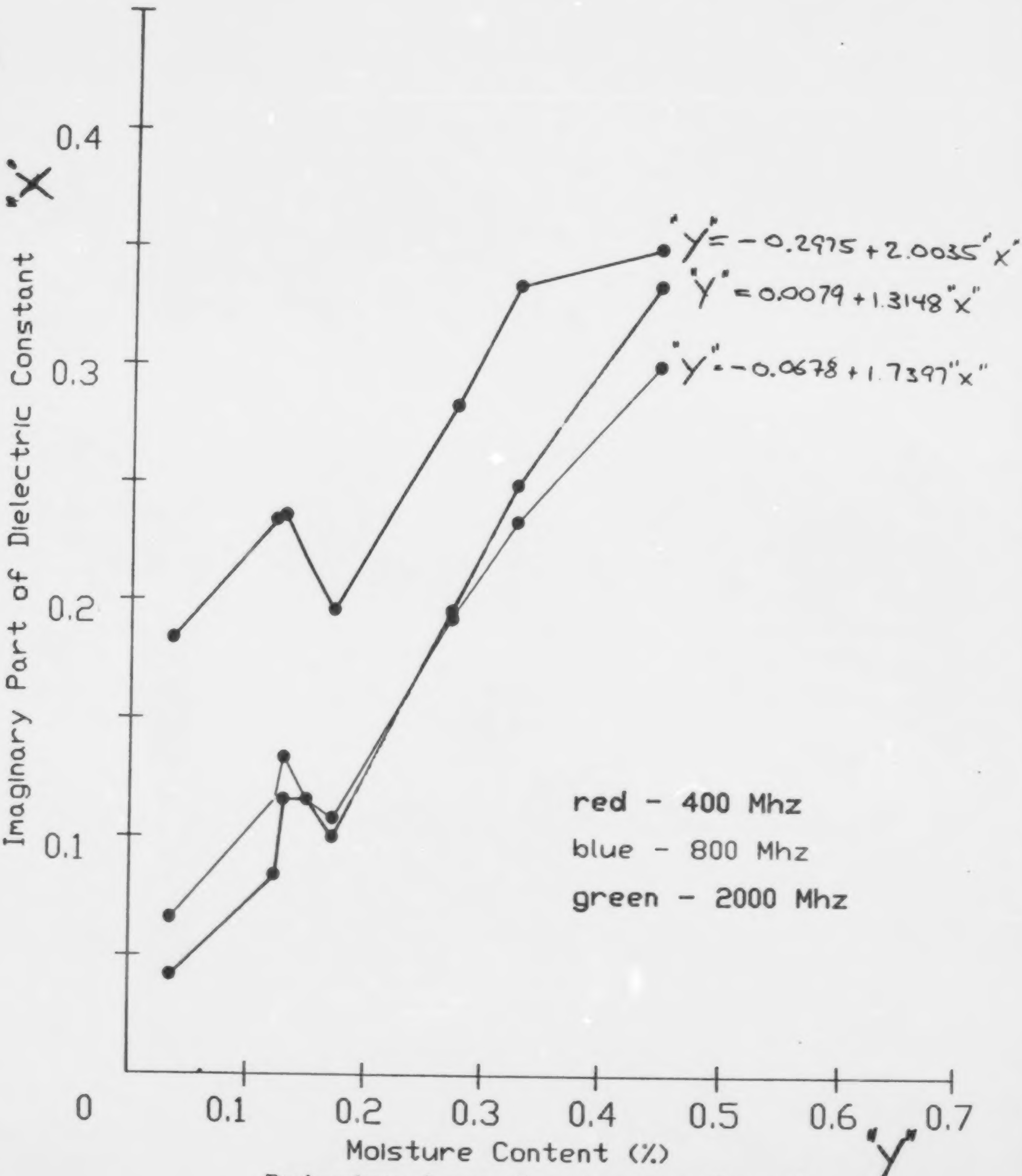
TABLE 1

The Dielectric Loss Measurements for Lanigan Granular
(no treatment)

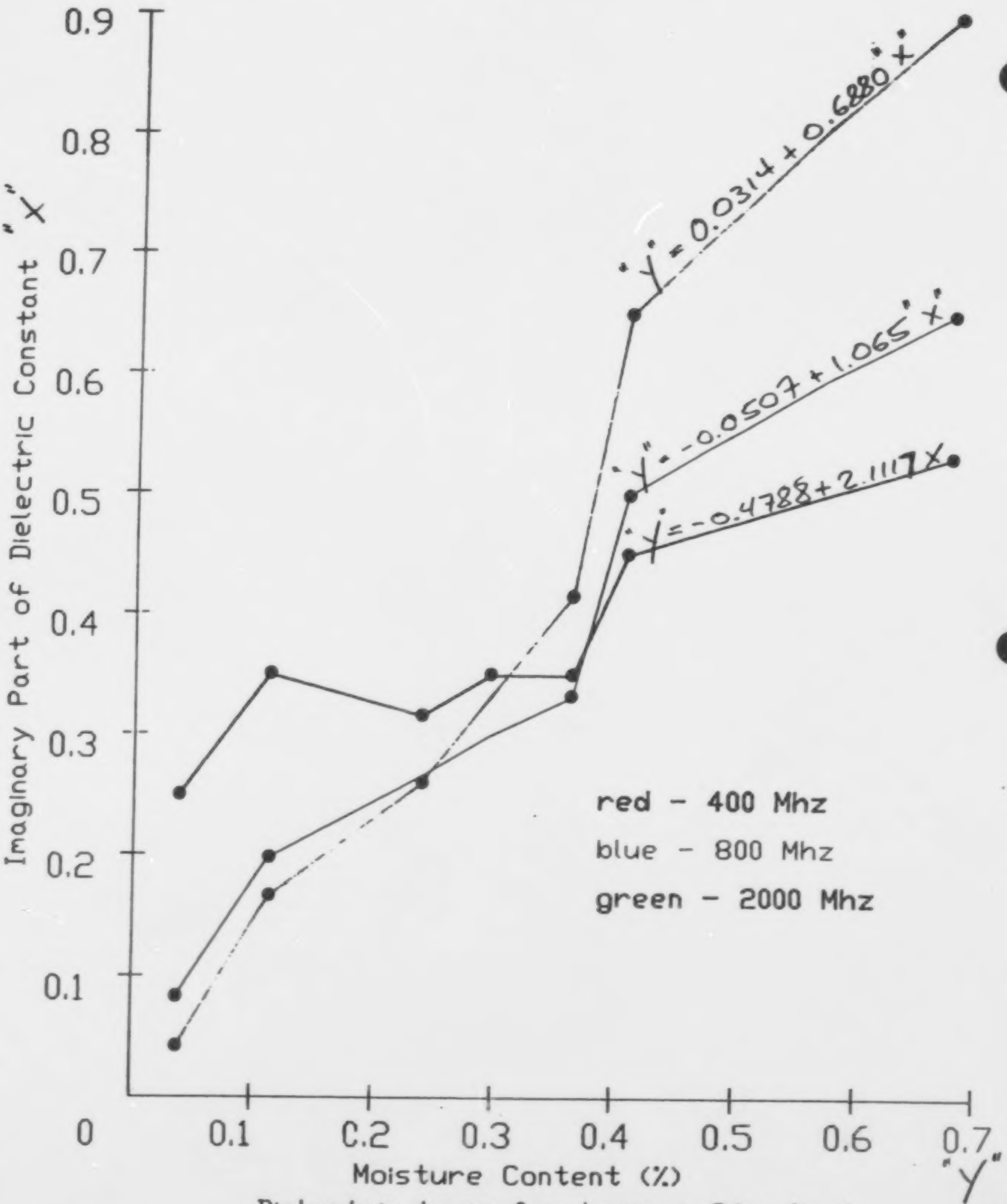
Moisture Freq	0.0377%	0.113%	0.240%	0.297%	0.366%	0.411%	0.572%	0.679%
400 Mhz	0.04	0.17	0.26	0.33	0.42	0.65	0.81	0.90
800 Mhz	0.08	0.20	0.27	0.30	0.33	0.50	0.60	0.65
2000 Mhz	0.25	0.35	0.32	0.35	0.35	0.45	0.50	0.53

TABLE 2

The Dielectric Loss Measurements for Lanigan Standard
(finer particle size - no treatment)



Dielectric Loss for Lanigan Granular



Dielectric Loss for Lanigan Standard

APPENDIX IV

MONITORING RESULTS AND GRAPHS

FIRST MONITORING PERIOD

CLIMATE CHAMBER EXPERIMENT # 1

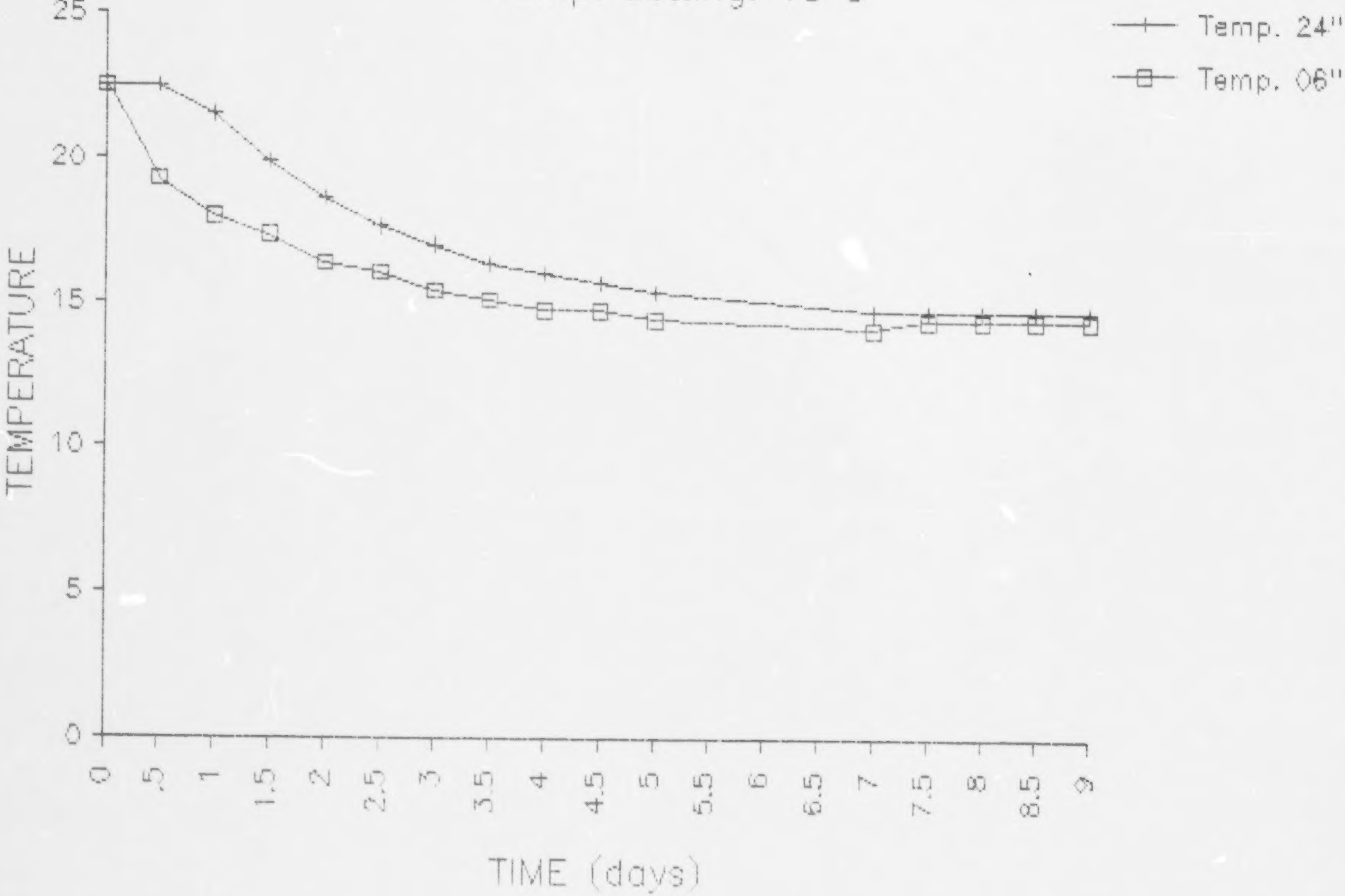
Operating Conditions: Temp. 15 c
R. H. 50-55 %

Product: Cory 210

Date	Time	DAYS	Temp. 24"	% H20 24"	R.H. 24"	Temp. 06"	% H20 06"	R.H. 06"	%H20 TOP
03-07-88	0	0	22.5	.039	12	22.5	.035	12	.037
	12	.5	22.5		10.4	19.3		14.6	
03-08-88	24	1	21.54	.04	10.8	18.02	.044	17.6	
	36	1.5	19.94		11.8	17.38		20.4	
03-09-88	48	2	18.66	.047	13.8	16.42	.054	23	.082
	60	2.5	17.7		16	16.1		25.4	
03-10-88	72	3	17.06	.06	18.4	15.46	.059	28	.07
	84	3.5	16.42		20.8	15.14		30.6	
03-11-88	96	4	16.1	.067	23.2	14.82	.07	32.8	.089
	108	4.5	15.78		25.2	14.82		34.2	
03-12-88	120	5	15.46		27.2	14.5		35.6	
	132	5.5							
03-13-88	144	6							
	156	6.5							
03-14-88	168	7	14.82	.054	33.2	14.18	.056	39.2	.07
	180	7.5	14.82		34.2	14.5		39.8	
03-15-88	192	8	14.82	.079	35.2	14.5	.079	40.4	.079
	204	8.5	14.82		36.2	14.5		40.8	
03-16-88	216	9	14.82	.089	37	14.5	.074	41.4	.084

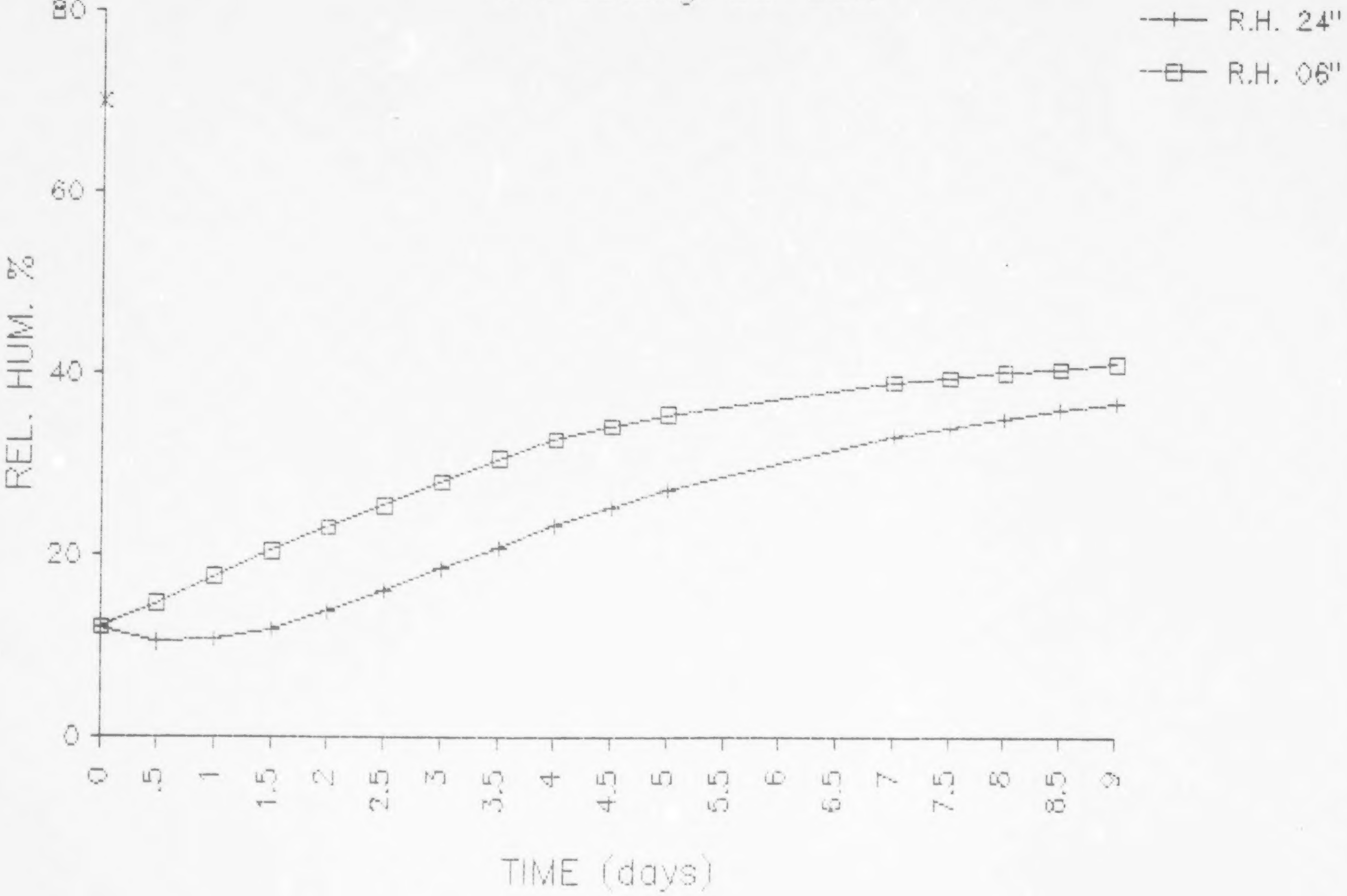
TEMPERATURE CHANGE/TIME

Temp. setting: 15 C



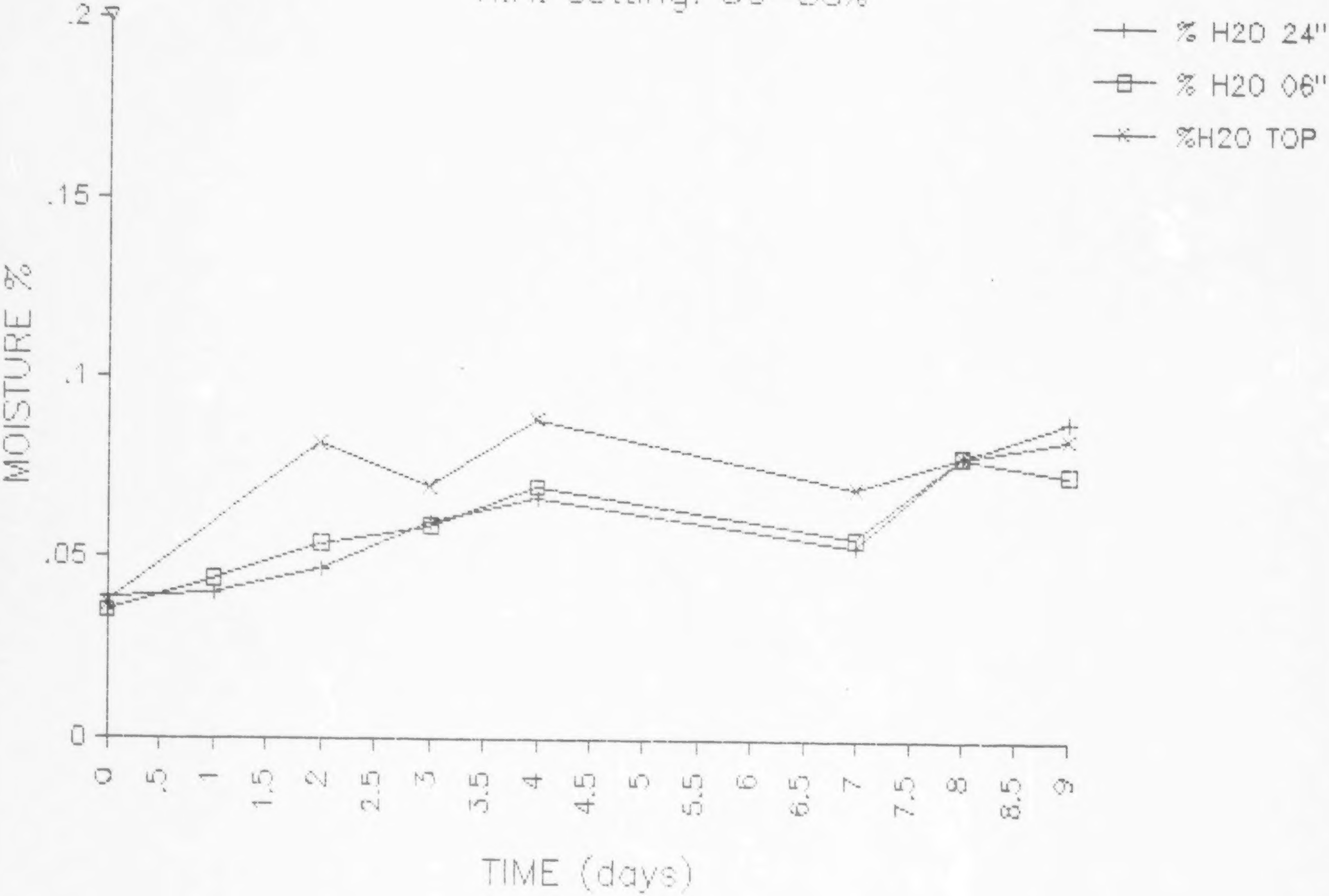
RELATIVE HUMIDITY CHANGE/TIME

R.H. setting: 50—55%



MOISTURE CHANGE/TIME

R.H. setting: 50–55%



CLIMATE CHAMBER EXPERIMENT # 1

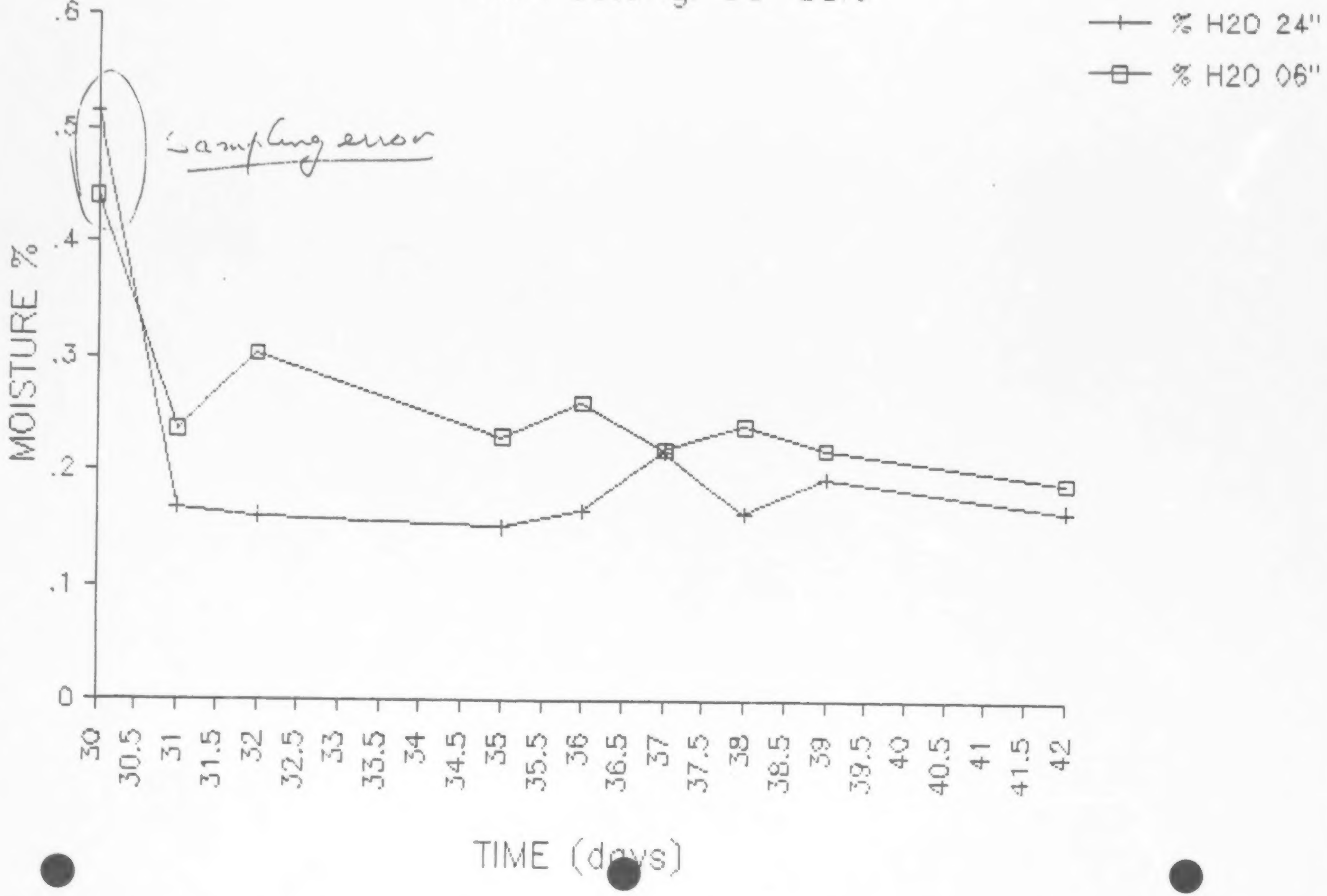
Operating Conditions: Temp. 15 c
R. H. 70-75 %

Product: Cory 210

Date	Time	DAYS	Temp. 24"	% H20 24"	R.H. 24"	Temp. 06"	% H20 06"	R.H. 06"	%H20 TOP
03-16-88	216	9	14.82	.089	37	14.5	.074	41.4	.084
	228	9.5	14.82		37.6	14.5		42.2	
03-17-88	240	10	14.82	.073	38.2	14.82	.075	43.4	.122
	252	10.5	14.82		39	14.82		44.2	
03-18-88	264	11	15.14	.071	39.6	14.82	.076	44.6	.125
	276	11.5	15.14		40.4	14.82		45.2	
03-19-88	288	12	15.14		41	14.82		45.4	
	300	12.5	15.14		41.6	14.82		45.8	
03-20-88	312	13	14.82		42.2	14.82		46	
	324	13.5	14.82		42.6	14.82		46.2	
03-21-88	336	14	15.14	.096	43	14.5	.095	46.6	.148
	348	14.5	14.82		43.4	15.46		47	
03-22-88	360	15	15.46	.08	43.8	15.14	.092	47.2	.151
	372	15.5	15.46		44.2	15.46		47.4	
03-23-88	384	16	15.46	.075	44.6	15.14	.089	47.8	.137
	396	16.5	15.46		44.8	15.46		48	
03-24-88	408	17	15.46	.092	45	14.82	.107	48.2	.165
	420	17.5	15.46		45.4	14.5		48.6	
03-25-88	432	18	15.14	.092	45.4	14.5	.116	48.6	.12

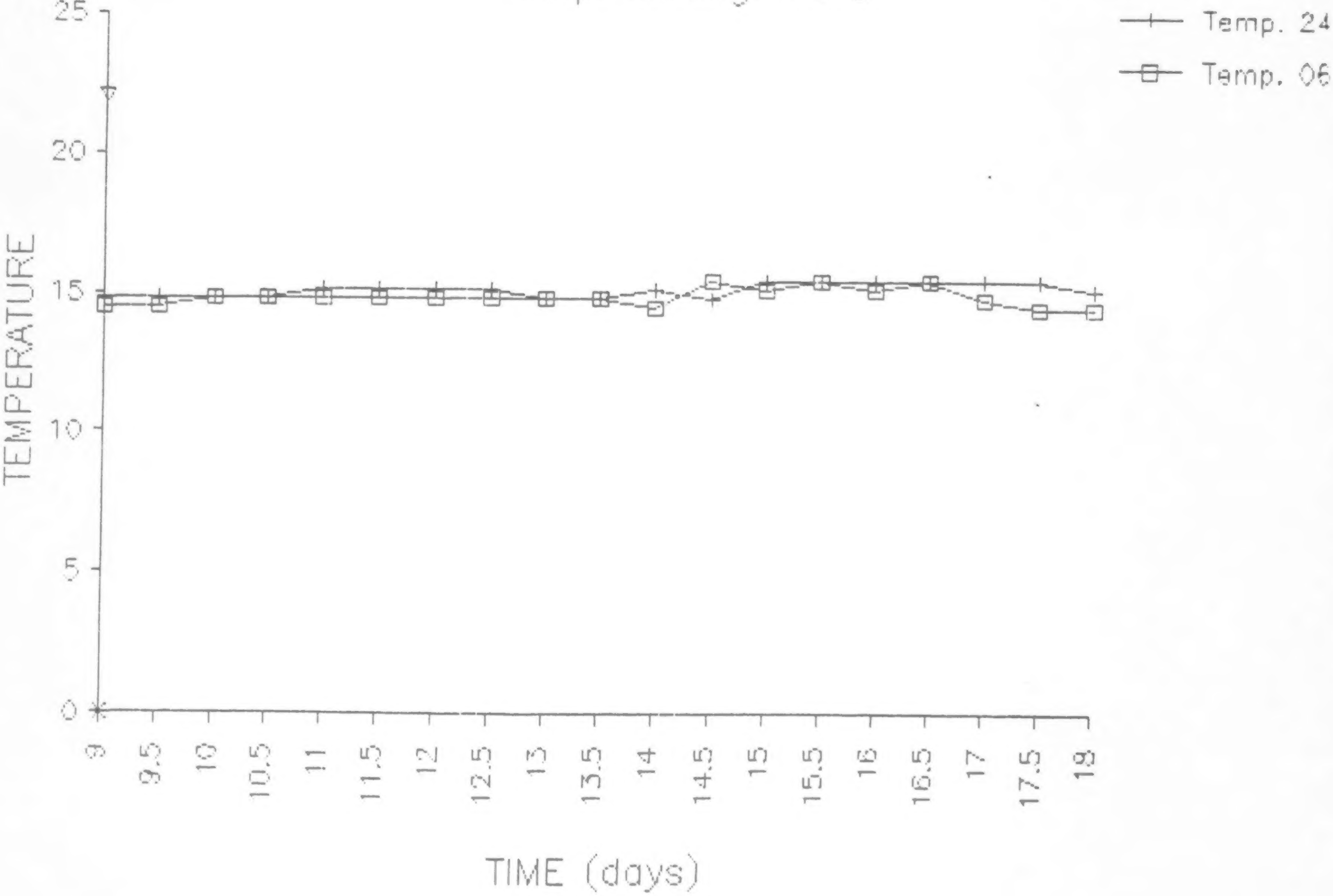
MOISTURE CHANGE/TIME

R.H. setting: 50–55%



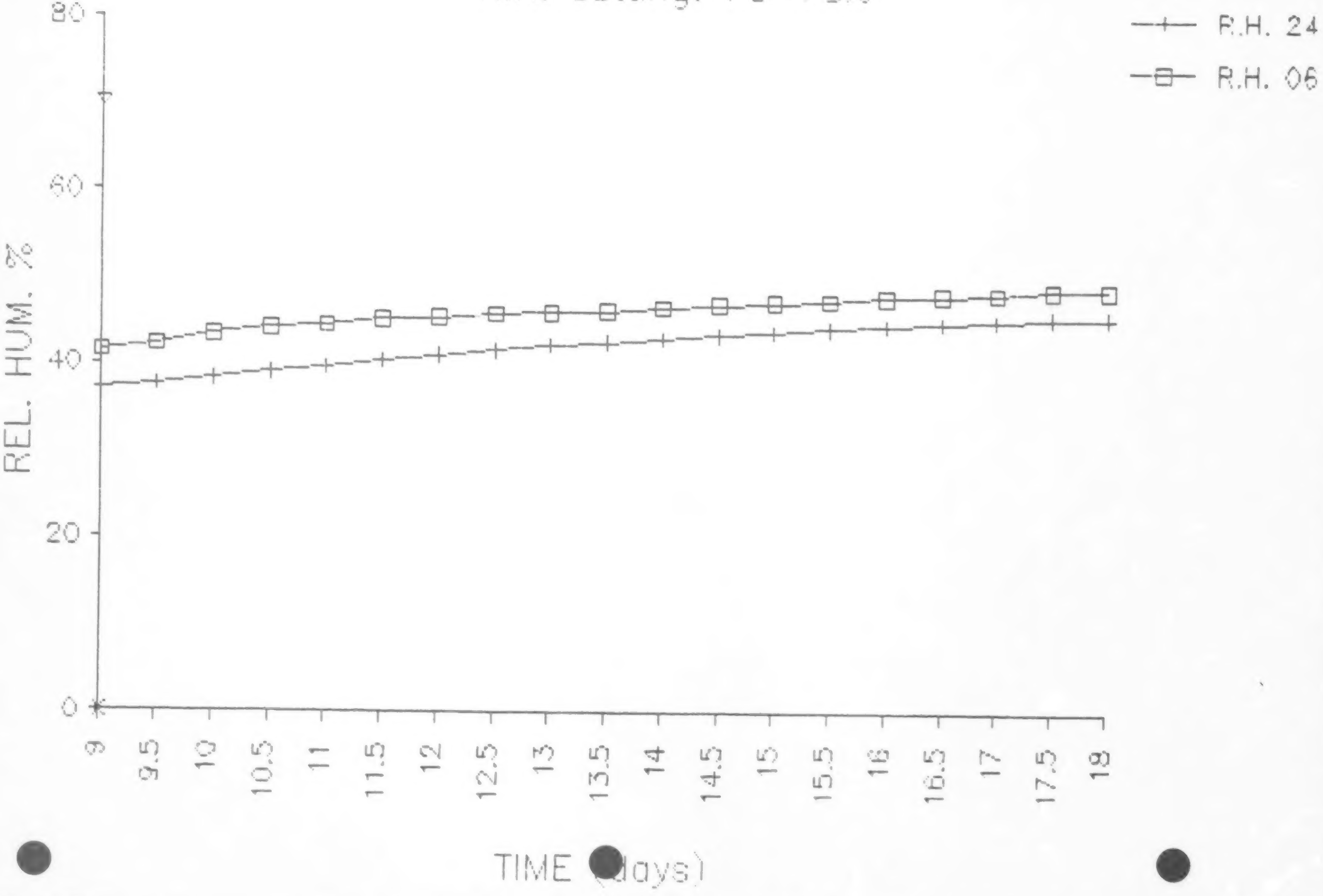
TEMPERATURE CHANGE/TIME

Temp. setting: 15 C



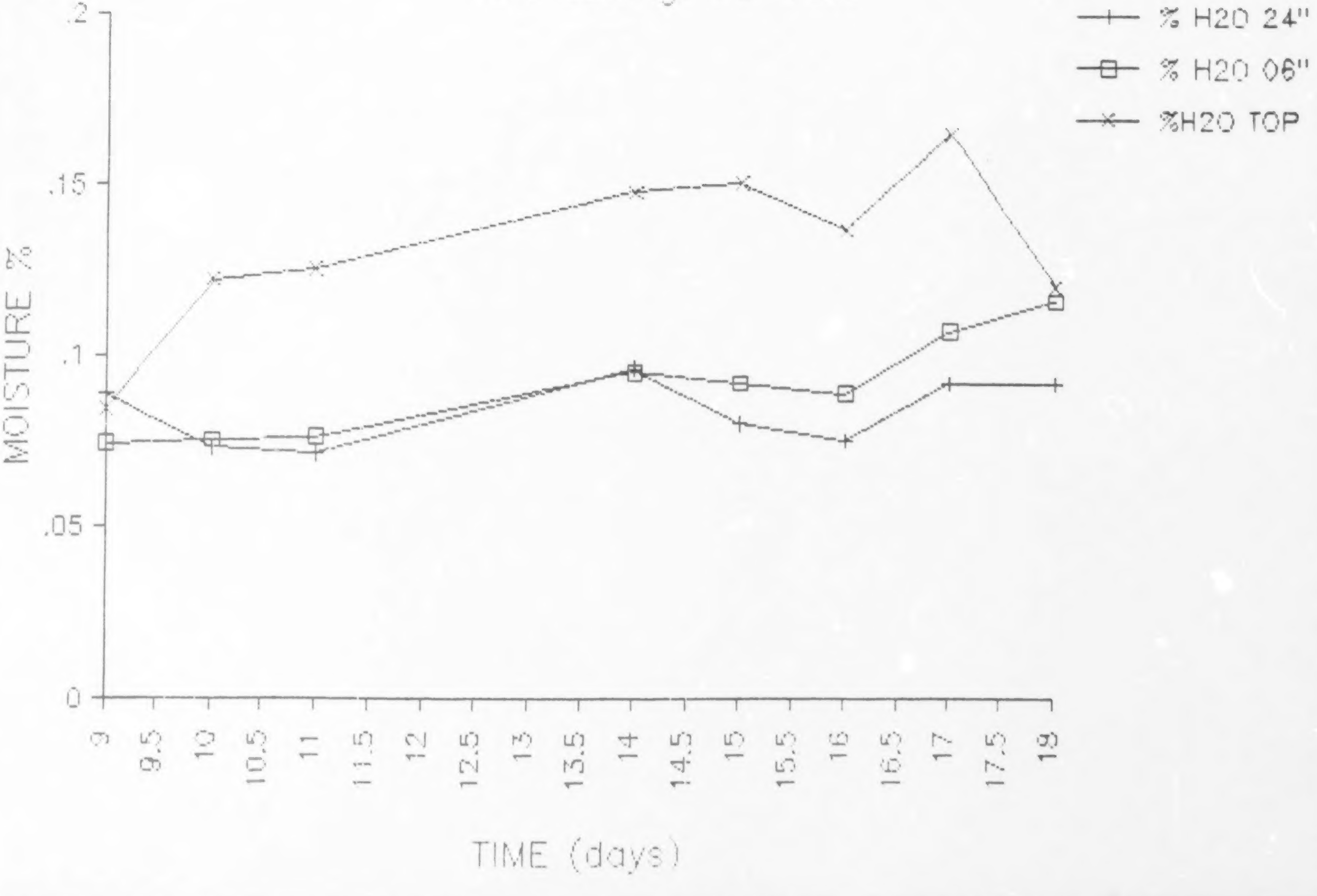
RELATIVE HUMIDITY CHANGE/TIME

R.H. setting: 70–75%



MOISTURE CHANGE/TIME

R.H. setting: 70–75%



CLIMATE CHAMBER EXPERIMENT # 1

Operating Conditions: Temp. 15 c
R. H. 85-90%

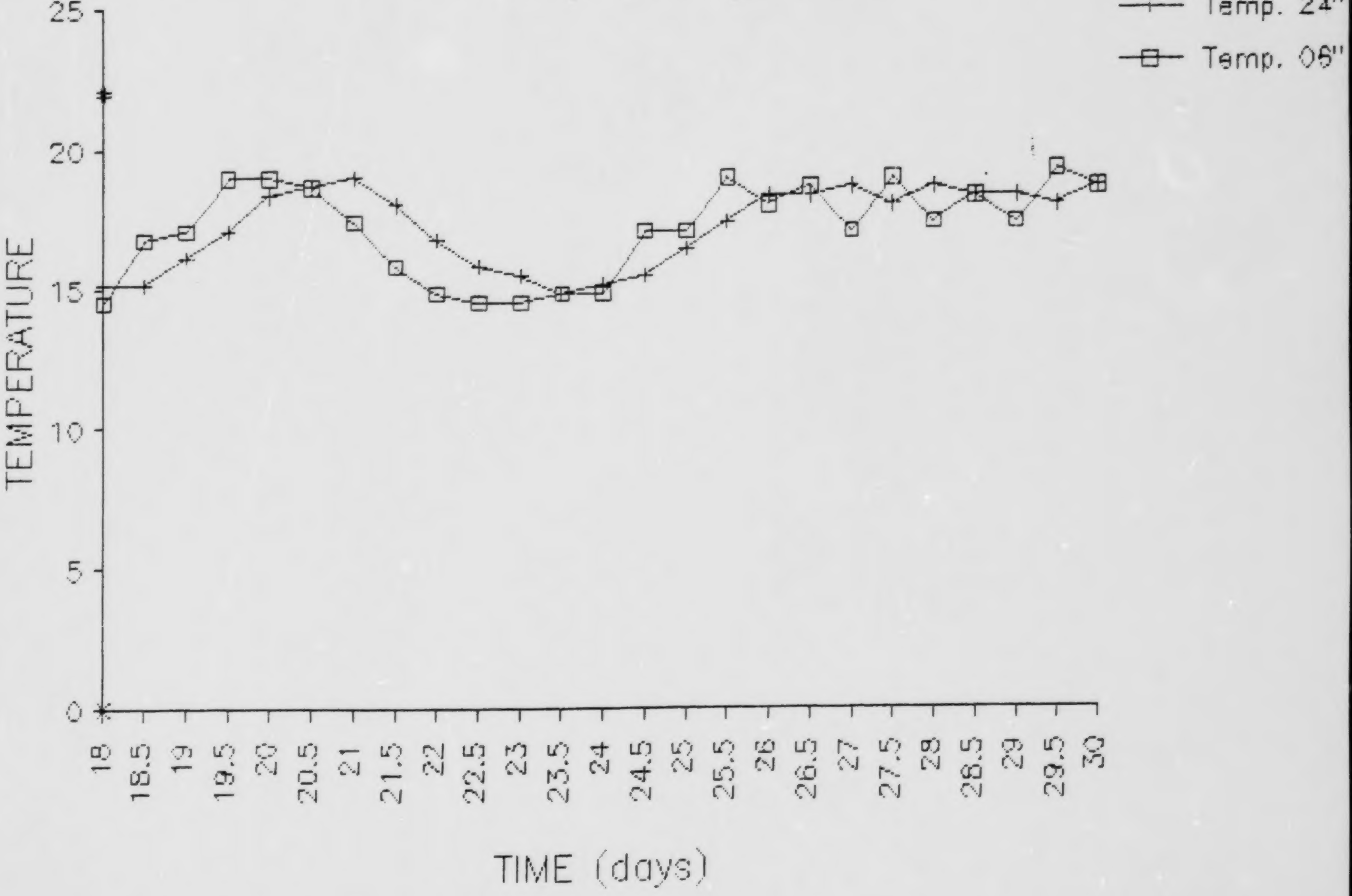
Product: Cory 210

Date	Time	DAYS	Temp. 24"	% H20 24"	R.H. 24"	Temp. 06"	% H20 06"	R.H. 06"	%H20 TOP
03-25-88	432	18	15.14	.092	45.4	14.5	.116	48.6	.12
	444	18.5	15.14		45.6	16.74		48.8	
03-26-88	456	19	16.1		46	17.06		49.2	
	468	19.5	17.06		46.2	18.98		49.8	
03-27-88	480	20	18.34		46.8	18.98		54.8	
	492	20.5	18.66		52.6	18.66		59	
03-28-88	504	21	18.98	.088	56.4	17.38	.137	61	4.188
	516	21.5	18.02		58.2	15.78		62	
03-29-88	528	22	16.74	.086	59	14.82	.138	62.6	1.292
	540	22.5	15.78		59.2	14.5		63.2	
03-30-88	552	23	15.46	.109	58	14.5	.145	63.6	.542
	564	23.5	14.82		56.8	14.82		63.8	
03-31-88	576	24	15.14	.13	56.2	14.82	.18	63.6	1.425
	588	24.5	15.46		56	17.06		63.2	
04-01-88	600	25	16.42		55.8	17.06		63	
	612	25.5	17.38		56	18.98		63	
04-02-88	624	26	18.34		56.2	18.02		62.8	
	636	26.5	18.34		56.4	18.66		62.8	
04-03-88	648	27	18.66		56.4	17.06		62.6	
	660	27.5	18.02		55.8	18.98		62.2	
04-04-88	672	28	18.66	.158	56	17.38	.17	63.6	4.824
	684	28.5	18.34		60.2	18.34		64.4	
04-05-88	696	29	18.34	.146	61.8	17.38	.176	64.6	3.867
	708	29.5	18.02		62.2	19.3		64.8	
04-06-88	720	30	18.66	.514	62.4	18.66	.44	65	3.214

sampling error

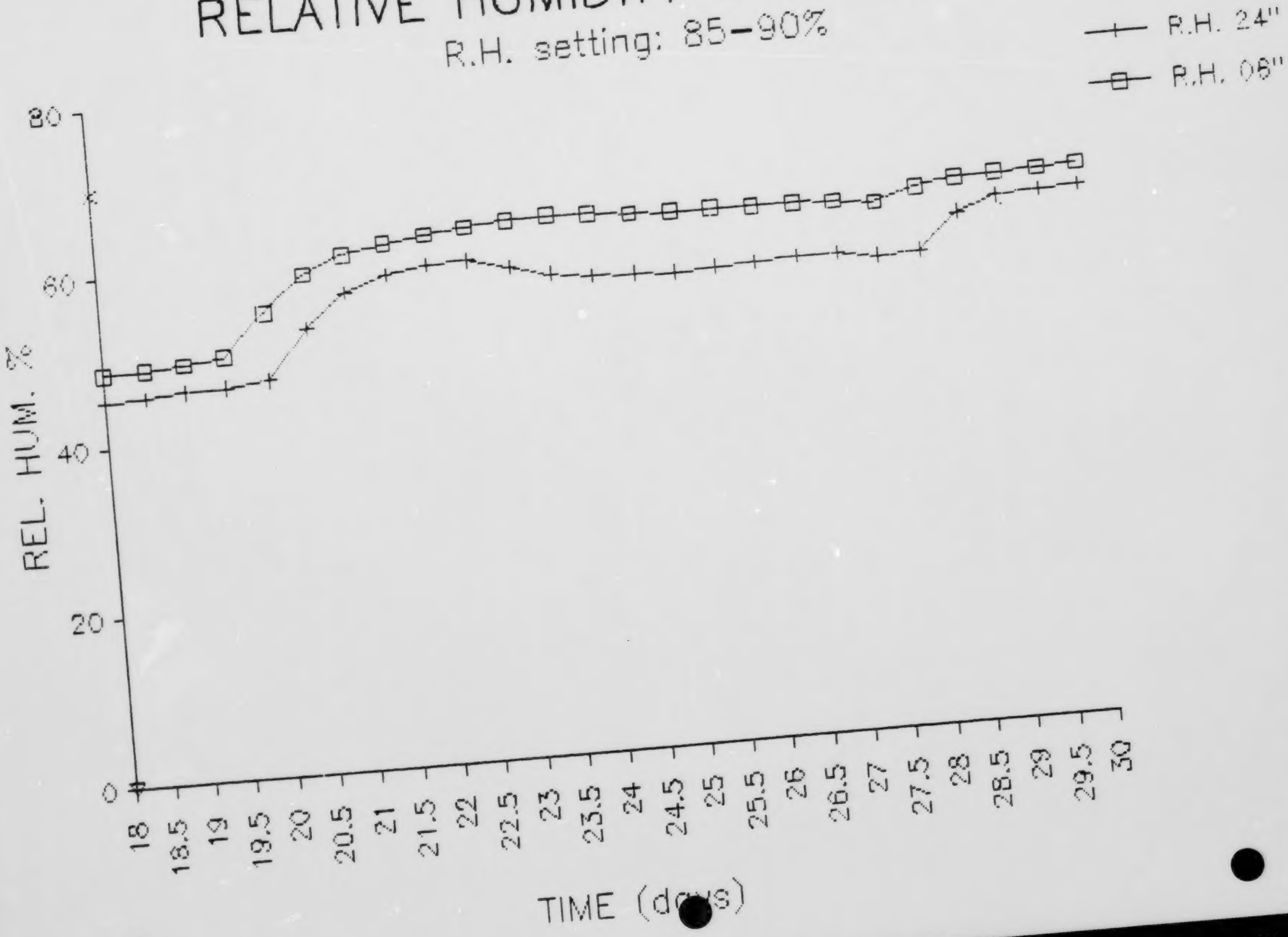
TEMPERATURE CHANGE/TIME

Temp. setting: 15 C



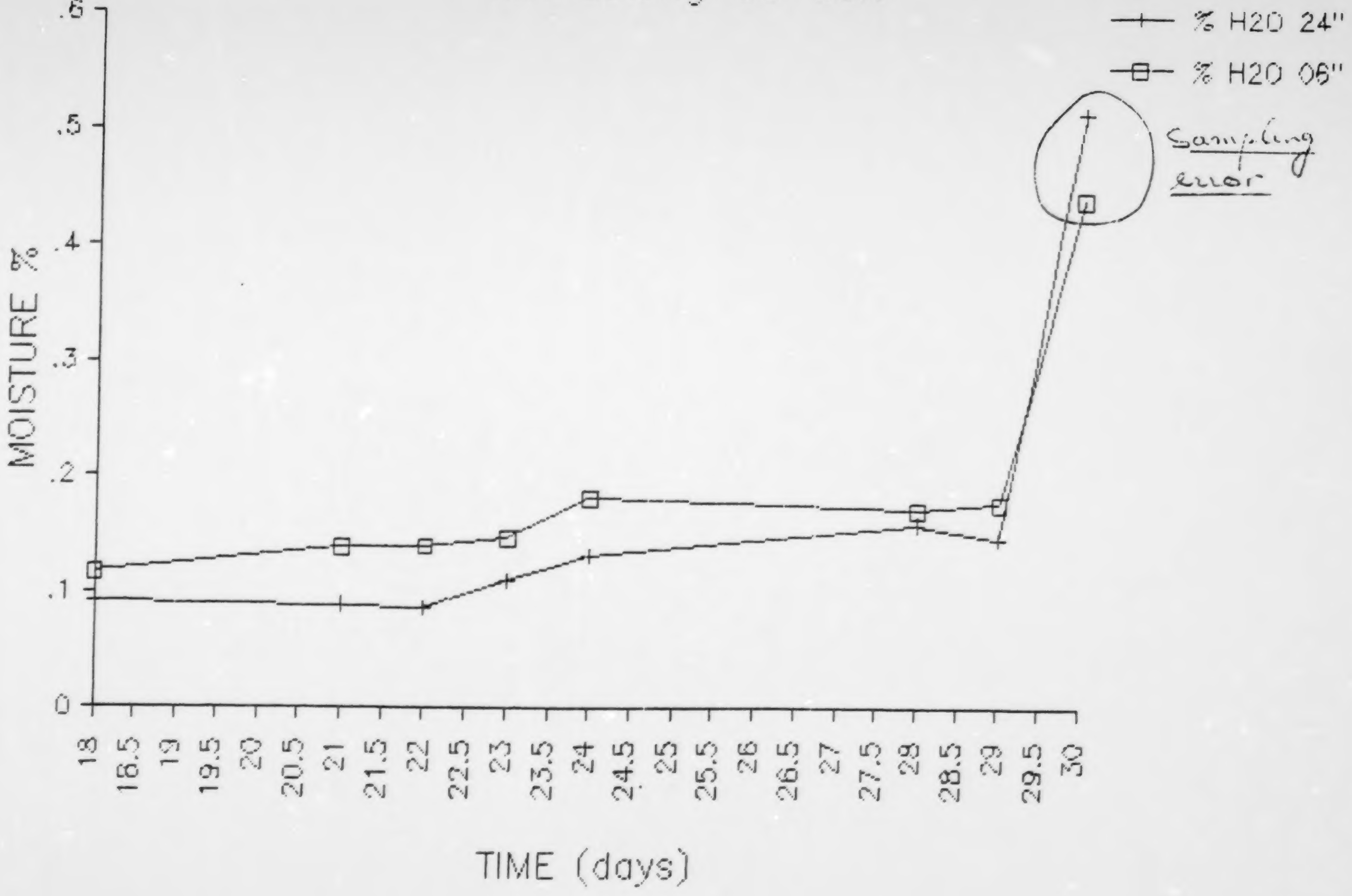
RELATIVE HUMIDITY CHANGE/TIME

R.H. setting: 85–90%

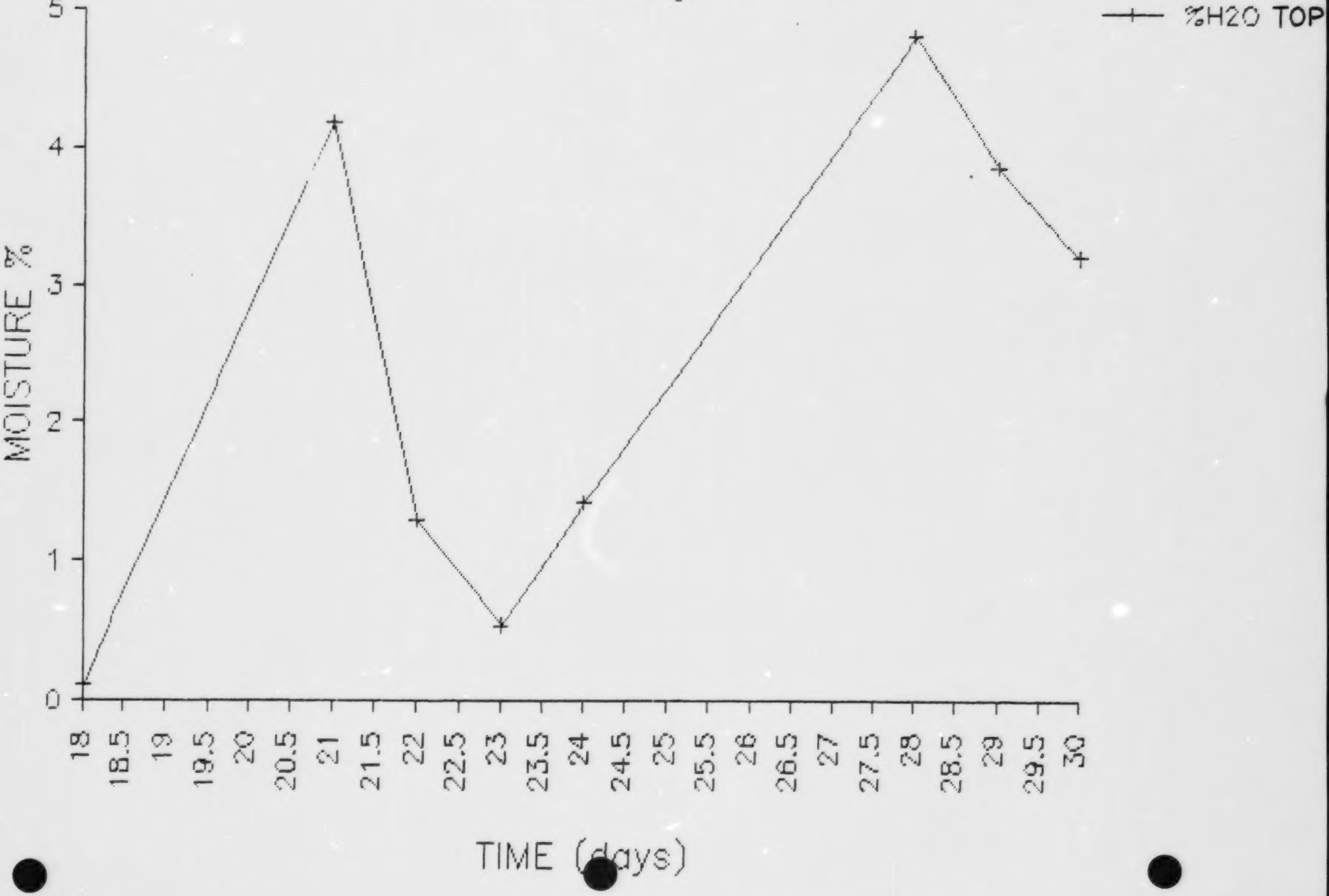


MOISTURE CHANGE/TIME

R.H. setting: 85–90%



Sampling error



MOISTURE CHANGE/TIME

R.H. setting: 85–90%

CLIMATE CHAMBER EXPERIMENT # 1

Operating Conditions: Temp. 15 c
R. H. 50-55%

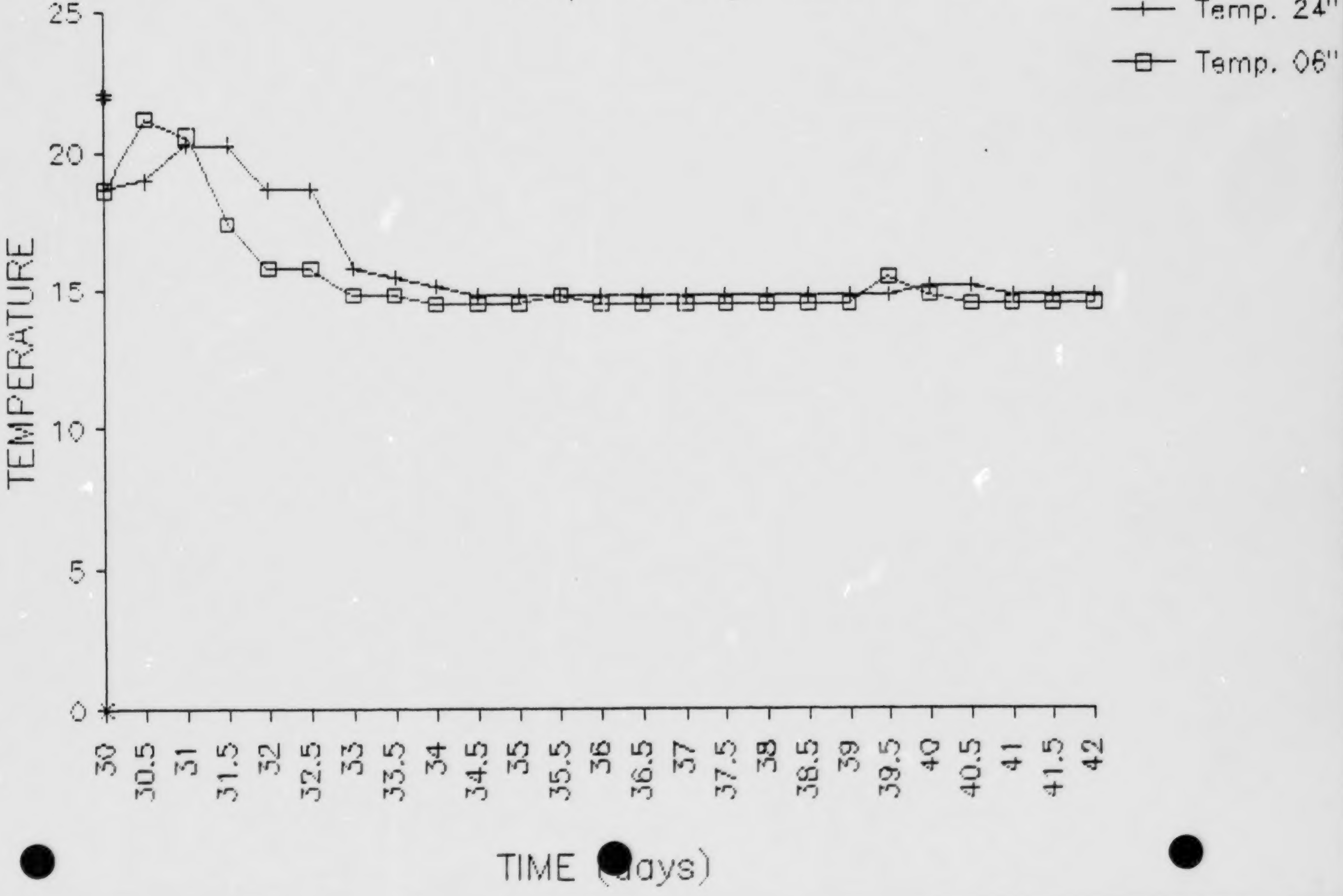
Product: Cory 210

Sampling error

Date	Time	DAYS	Temp. 24"	% H2O 24"	R.H. 24"	Temp. 06"	% H2O 06"	R.H. 06"	%H2O TOP
04-06-88	720	30	18.66	.514	62.4	18.66	.44	65	3.214
	732	30.5	18.98		62.4	21.22		65.2	
04-07-88	744	31	20.26	.168	63	20.58	.236	65.6	4.154
	756	31.5	20.26		63.4	17.38		65.8	
04-08-88	768	32	18.66	.16	63	15.78	.303	65.8	1.77
	780	32.5	18.66		63	15.78		65.8	
04-09-88	792	33	15.78		61	14.82		65.8	
	804	33.5	15.46		60.4	14.82		65.8	
04-10-88	816	34	15.14		60	14.5		66	
	828	34.5	14.82		59.8	14.5		65.8	
04-11-88	840	35	14.82	.151	59.8	14.5	.23	66	.237
	852	35.5	14.82		59.8	14.82		66	
04-12-88	864	36	14.82	.165	59.8	14.5	.26	66	.176
	876	36.5	14.82		59.8	14.5		66.2	
04-13-88	888	37	14.82	.218	59.8	14.5	.22	66.2	.128
	900	37.5	14.82		59.8	14.5		66.2	
04-14-88	912	38	14.82	.163	59.8	14.5	.24	66.2	.145
	924	38.5	14.82		59.8	14.5		66.2	
04-15-88	936	39	14.82	.194	59.8	14.5	.22	66.2	.12
	948	39.5	14.82		59.8	15.46		66.2	
04-16-88	960	40	15.14		59.8	14.82		66.2	
	972	40.5	15.14		59.8	14.5		66.2	
04-17-88	984	41	14.82		59.8	14.5		66.6	
	996	41.5	14.82		59.8	14.5		66.6	
04-18-88	1008	42	14.82	.166	59.8	14.5	.19	66.6	.116

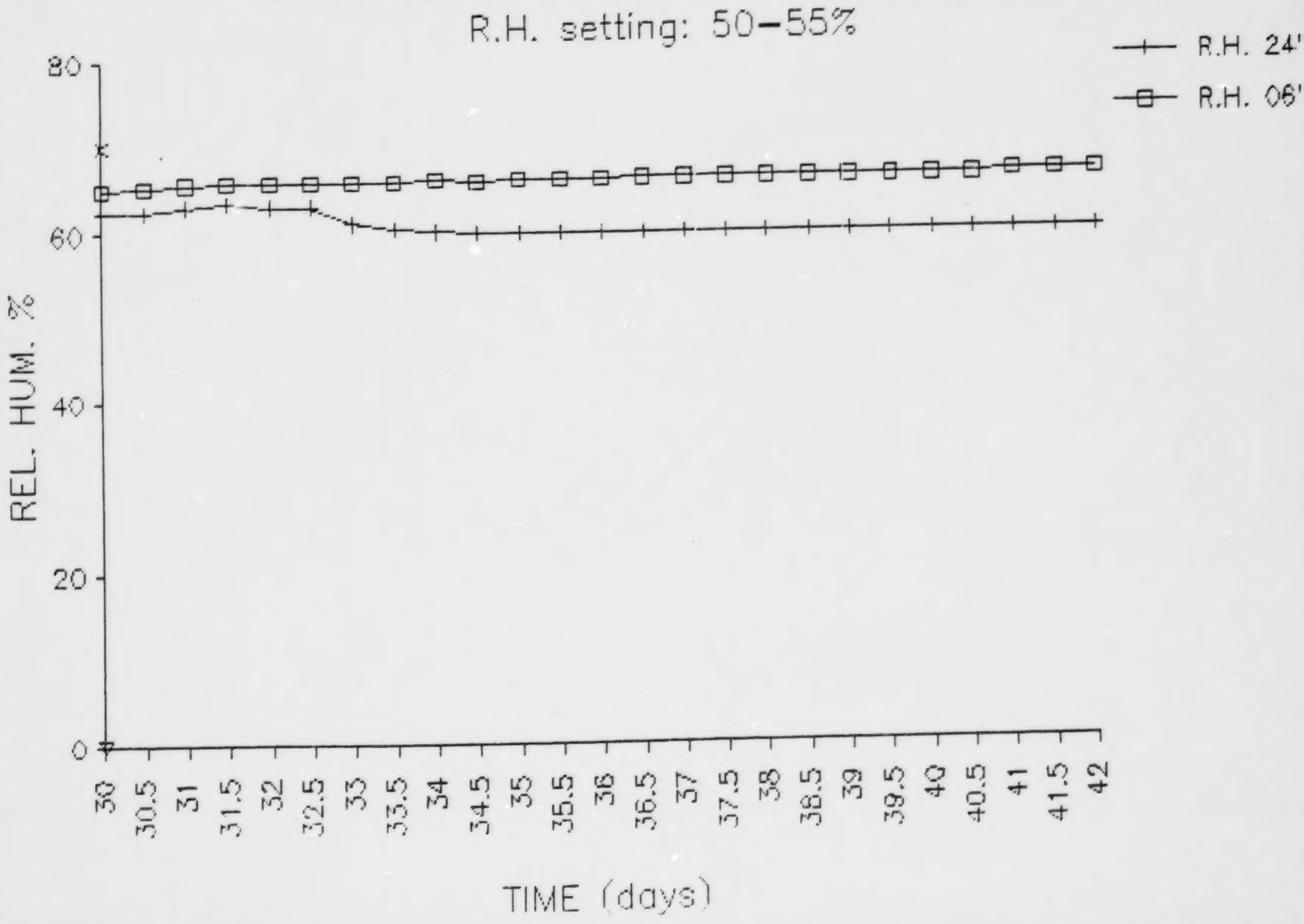
TEMPERATURE CHANGE/TIME

Temp. setting: 15 C



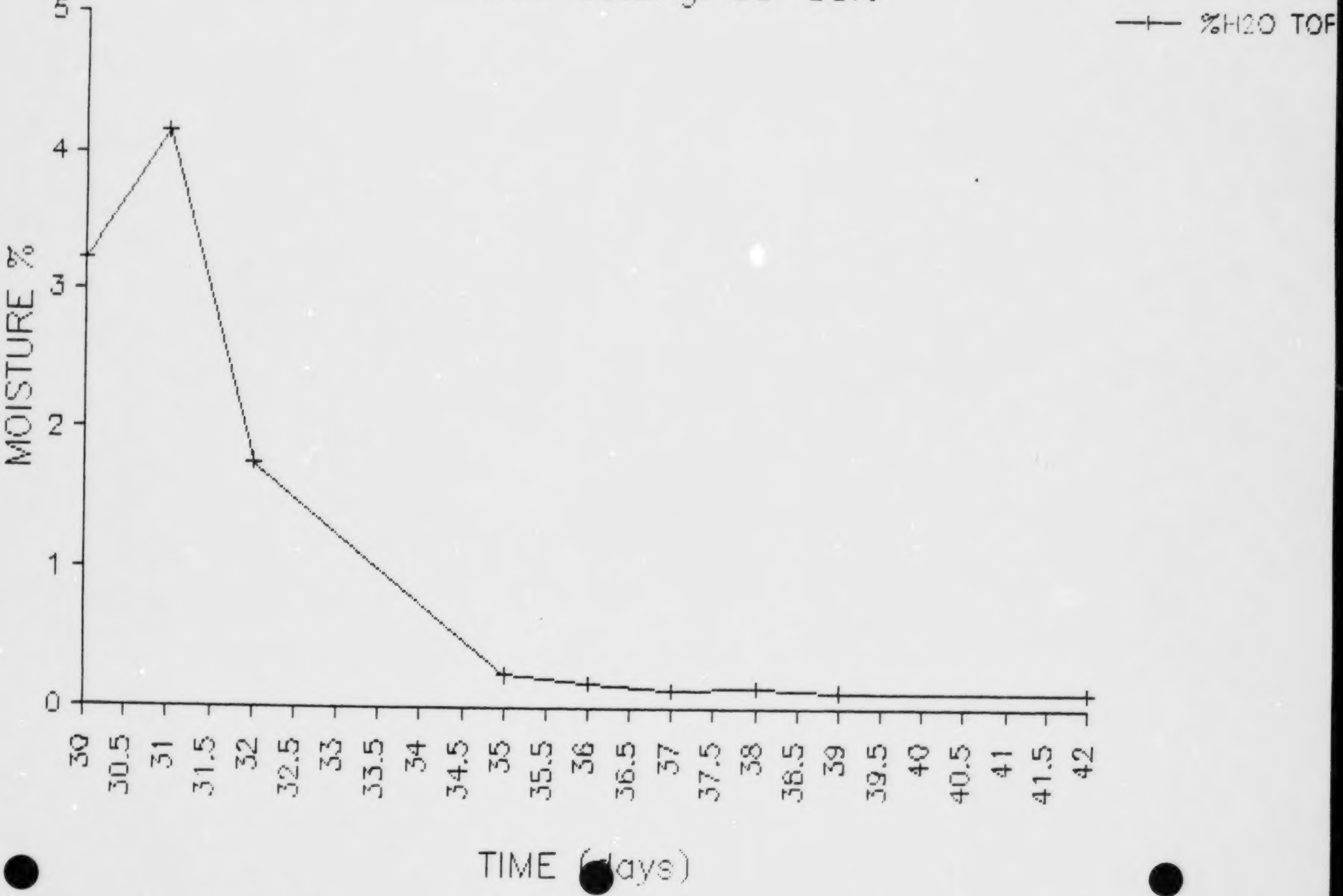
RELATIVE HUMIDITY CHANGE/TIME

R.H. setting: 50-55%



MOISTURE CHANGE/TIME

R.H. setting: 50–55%



APPENDIX V

MONITORING RESULTS AND GRAPHS

SECOND MONITORING PERIOD

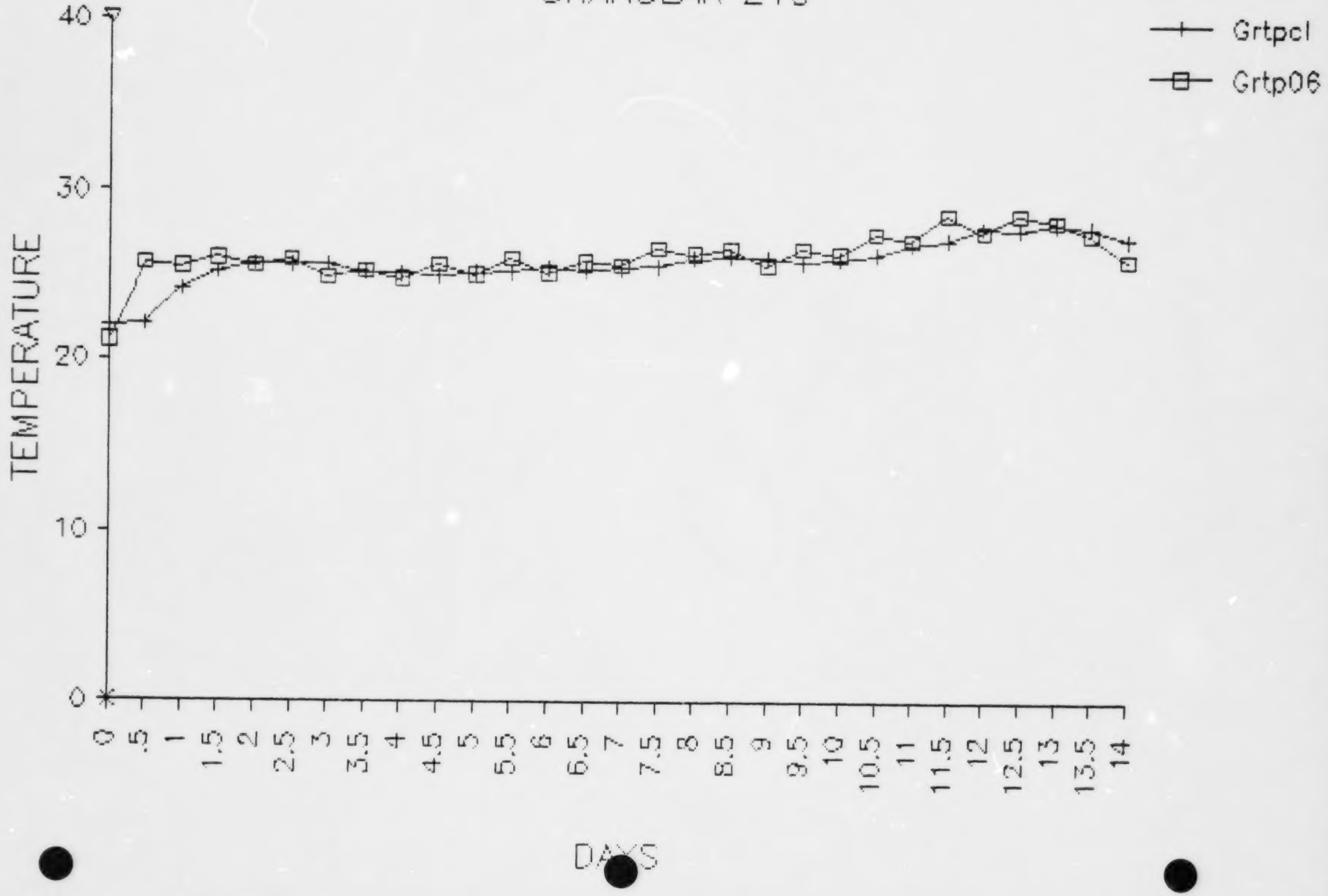
CLIMATE CHAMBER EXPERIMENT NO. 2

Setting Conditions: Temp. 25-30 C
R. H. 55-60%

Date	Time	DAYS	Grrh%cl	Grrh%06	GrH20%cl	GrH20%06	GrH20%top	Grtpcl	Grtp06
04-29-88	11:34:24	0	19.9	21.2	.067	.067	.067	21.866	21.072
04-30-88	0: 2:38	.5	27.5	38.8				21.987	25.613
	12: 2:38	1	34.3	41.2				24.059	25.439
05-01-88	0: 2:39	1.5	37.9	44.6				25.103	25.989
	12: 2:39	2	40.5	46				25.586	25.484
05-02-88	0: 2:40	2.5	42.7	47.6				25.601	25.885
	12: 2:40	3	44.3	48	.069	.084	.127	25.603	24.839
05-03-88	0: 2:40	3.5	45.5	49.4				25.176	25.261
	12: 2:41	4	46.9	49.8	.066	.069	.128	25.068	24.771
05-04-88	0: 2:42	4.5	48.1	50.6				24.933	25.654
	12: 2:42	5	49.1	50.8	.068	.07	.119	25.177	25.052
05-05-88	0: 2:43	5.5	49.9	51.8				25.183	26.034
	12: 2:43	6	50.5	51.8	.106	.098	.149	25.469	25.163
05-06-88	0: 2:43	6.5	50.9	52.2				25.297	25.846
	12:51:48	7	51.5	52.2	.091	.102	.112	25.461	25.635
05-07-88	0:51:48	7.5	51.7	52.6				25.59	26.692
	12:59:58	8	52.1	52.6				26.072	26.319
05-08-88	0:59:58	8.5	52.5	53				26.194	26.673
	12:59:59	9	52.7	53				26.148	25.643
05-09-88	0:59:59	9.5	52.7	53.2				25.838	26.673
	12: 0: 0	10	53.1	53.4				26.08	26.347
05-10-88	0: 0: 0	10.5	53.3	53.6				26.335	27.578
	12: 0: 1	11	53.5	53.6				26.936	27.205
05-11-88	0: 0: 1	11.5	53.5	53.8				27.245	28.735
	12: 0: 1	12	53.9	53.6	.085	.109	.116	27.948	27.705
05-12-88	0: 0: 2	12.5	53.9	53.8				27.822	28.751
	12: 0: 3	13	54.1	53.8	.08	.097	.116	28.164	28.324
05-13-88	0: 0: 3	13.5	54.1	53.8				28.01	27.599
	12:47: 3	14	54.1	53.8	.103	.099	.137	27.266	26.052

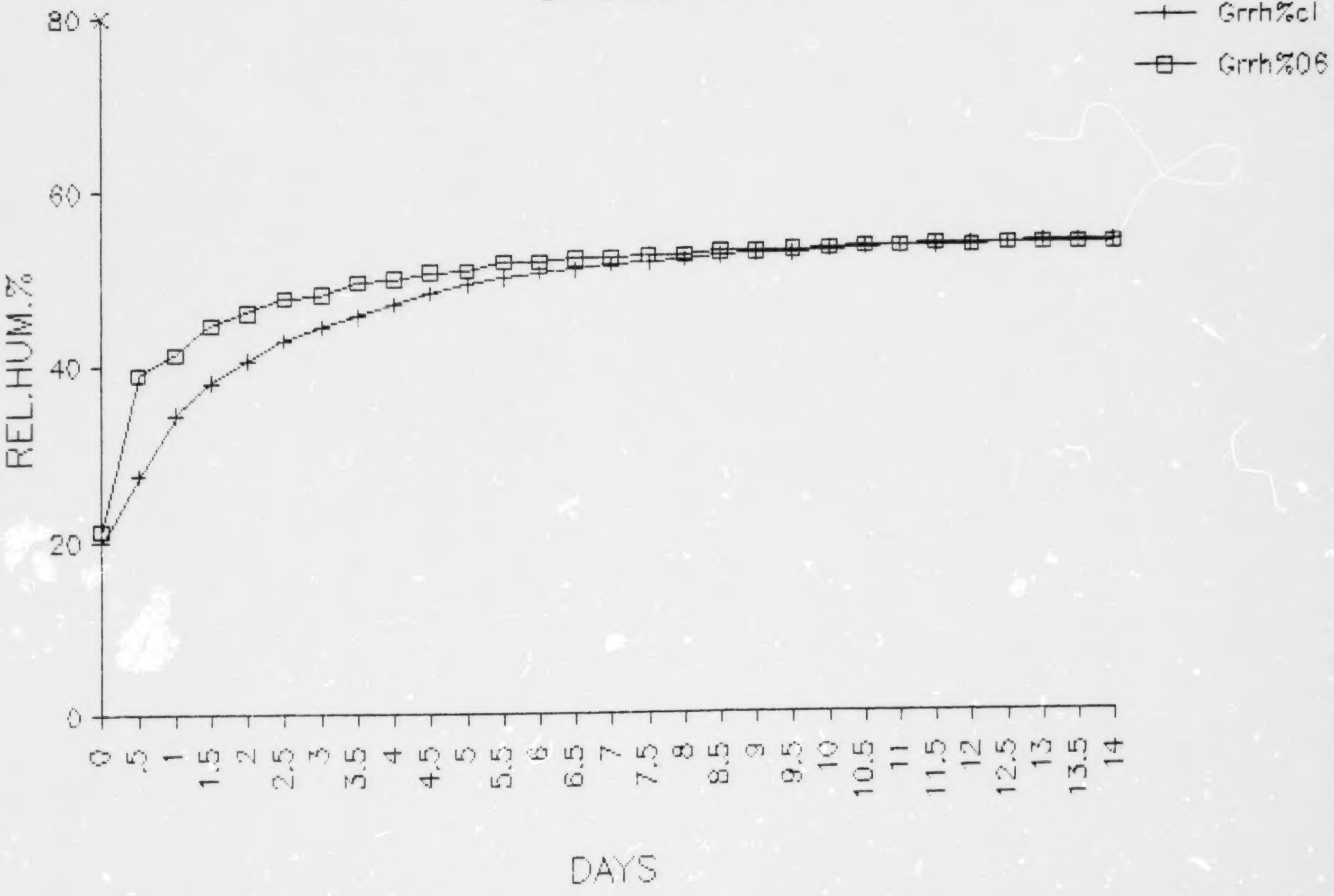
TEMPERATURE (setting:25-30 C)

GRANULAR 210



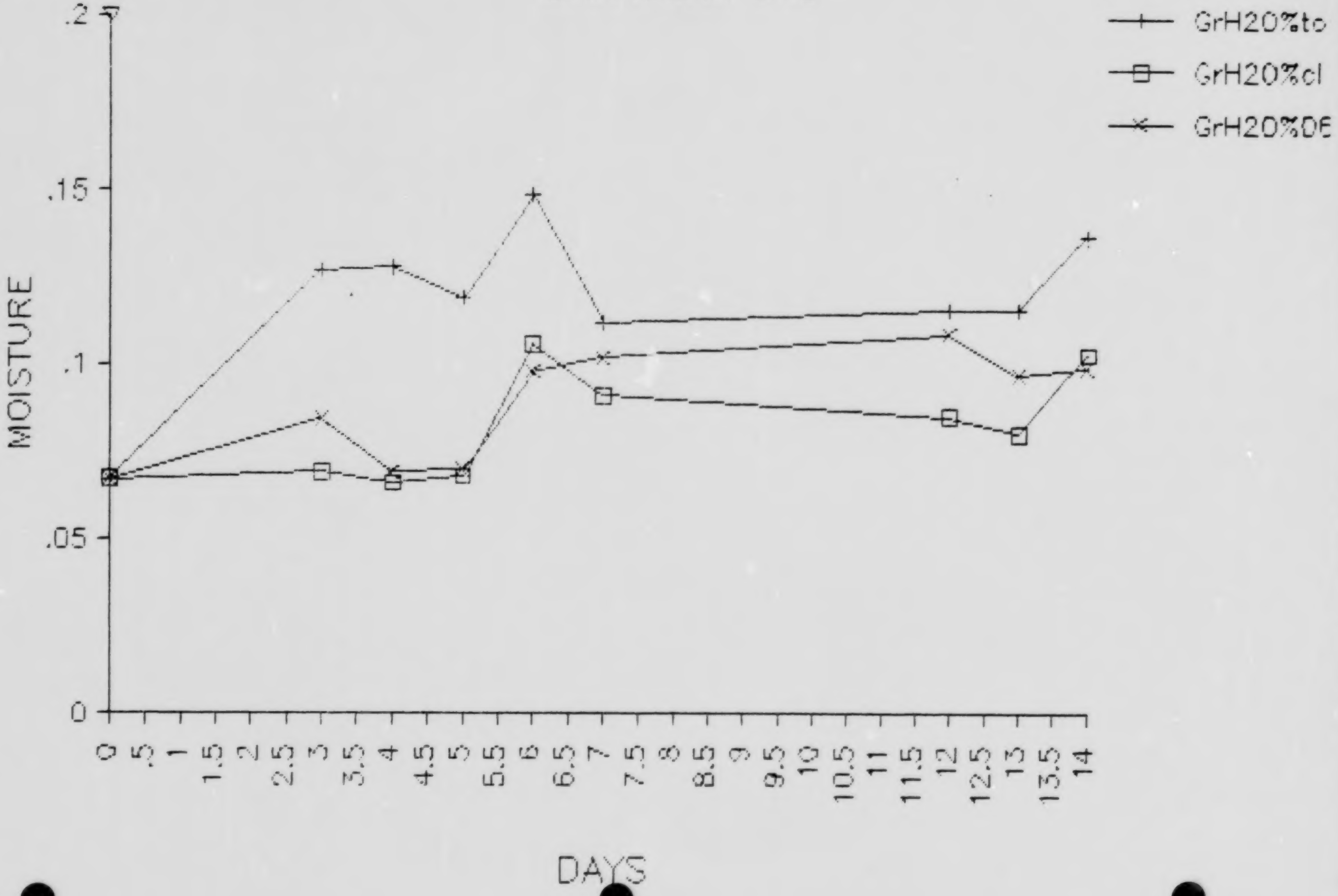
REL. HUM. (setting:55-60%)

GRANULAR 210



MOISTURE

GRANULAR 210



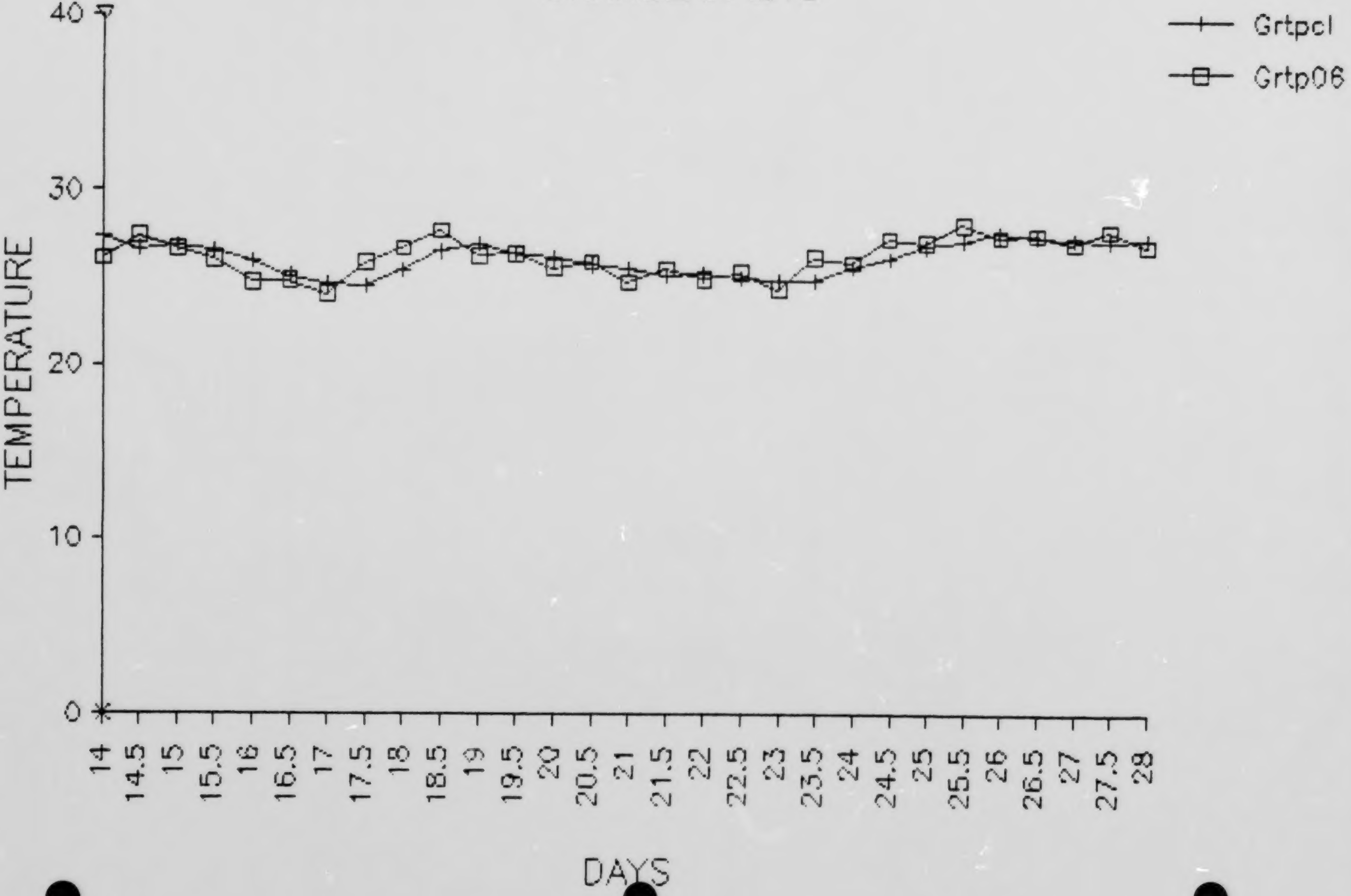
CLIMATE CHAMBER EXPERIMENT NO. 2

Setting Conditions: Temp. 25-30 C
R. H. 70%

Date	Time	DAYS	Grrh%cl	Grrh%06	GrH20%cl	GrH20%06	GrH20%top	Grtpcl	Grtp06
05-13-88	12:47: 3	14	54.1	53.8	.103	.099	.137	27.266	26.052
05-14-88	0:47: 4	14.5	54.1	54.4				26.588	27.383
	12:47: 4	15	54.3	54.6				26.781	26.629
05-15-88	0:47: 5	15.5	54.3	55.2				26.49	25.987
	12:47: 5	16	54.5	55.6				25.883	24.683
05-16-88	0:47: 6	16.5	54.5	56.2				25.023	24.767
	12:47: 6	17	54.5	56.8	.084	.126	.225	24.562	24.011
05-17-88	0:47: 7	17.5	54.7	57.8				24.451	25.814
	12:47: 7	18	54.7	58.6	.088	.129	.377	25.379	26.689
05-18-88	0:47: 8	18.5	54.9	59.4				26.487	27.723
	12:47: 8	19	55.1	59.6	.113	.146	.278	26.944	26.231
05-19-88	0:47: 9	19.5	55.1	60.2				26.408	26.363
	12:48:58	20	55.3	60.4	.111	.136	.264	26.091	25.548
05-20-88	0:48:59	20.5	55.5	61				25.728	25.887
	12:48:59	21	55.5	61				25.509	24.749
05-21-88	0:49: 0	21.5	55.7	61.8				25.087	25.492
	12:49: 0	22	55.9	62				25.199	24.927
05-22-88	0:49: 0	22.5	56.1	62.4				25.021	25.285
	12:49: 1	23	56.3	62.4				24.884	24.378
05-23-88	0:49: 1	23.5	56.5	63.2				24.857	26.202
	12:49: 2	24	56.7	63.2				25.615	25.911
05-24-88	0:49: 2	24.5	57.3	63.8				26.127	27.345
	12:49: 3	25	57.7	63.8	.099	.132	.264	26.869	27.097
05-25-88	0:49: 3	25.5	58.5	64.2				27.184	28.16
	12:48: 4	26	59.3	64.2	.128	.141	.293	27.662	27.43
05-26-88	0:48: 4	26.5	59.9	64.6				27.415	27.6
	12:48: 5	27	60.3	64.6	.144	.159	.379	27.288	27.066
05-27-88	0:48: 5	27.5	60.9	65				27.238	27.83
	10:48: 5	28	61.3	64.8	.122	.145	.347	27.341	26.92

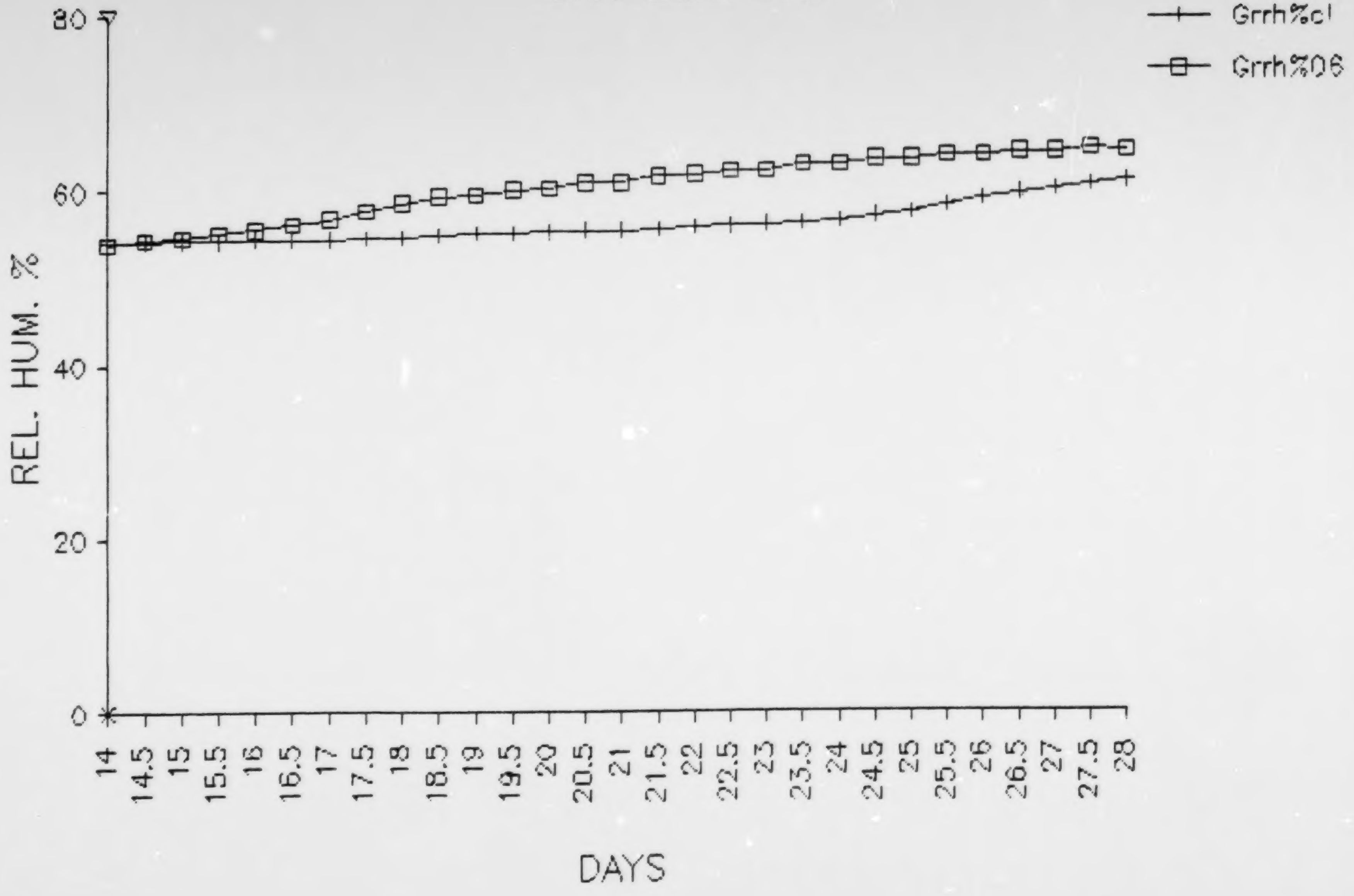
TEMPERATURE (setting:25-30 C)

GRANULAR 210



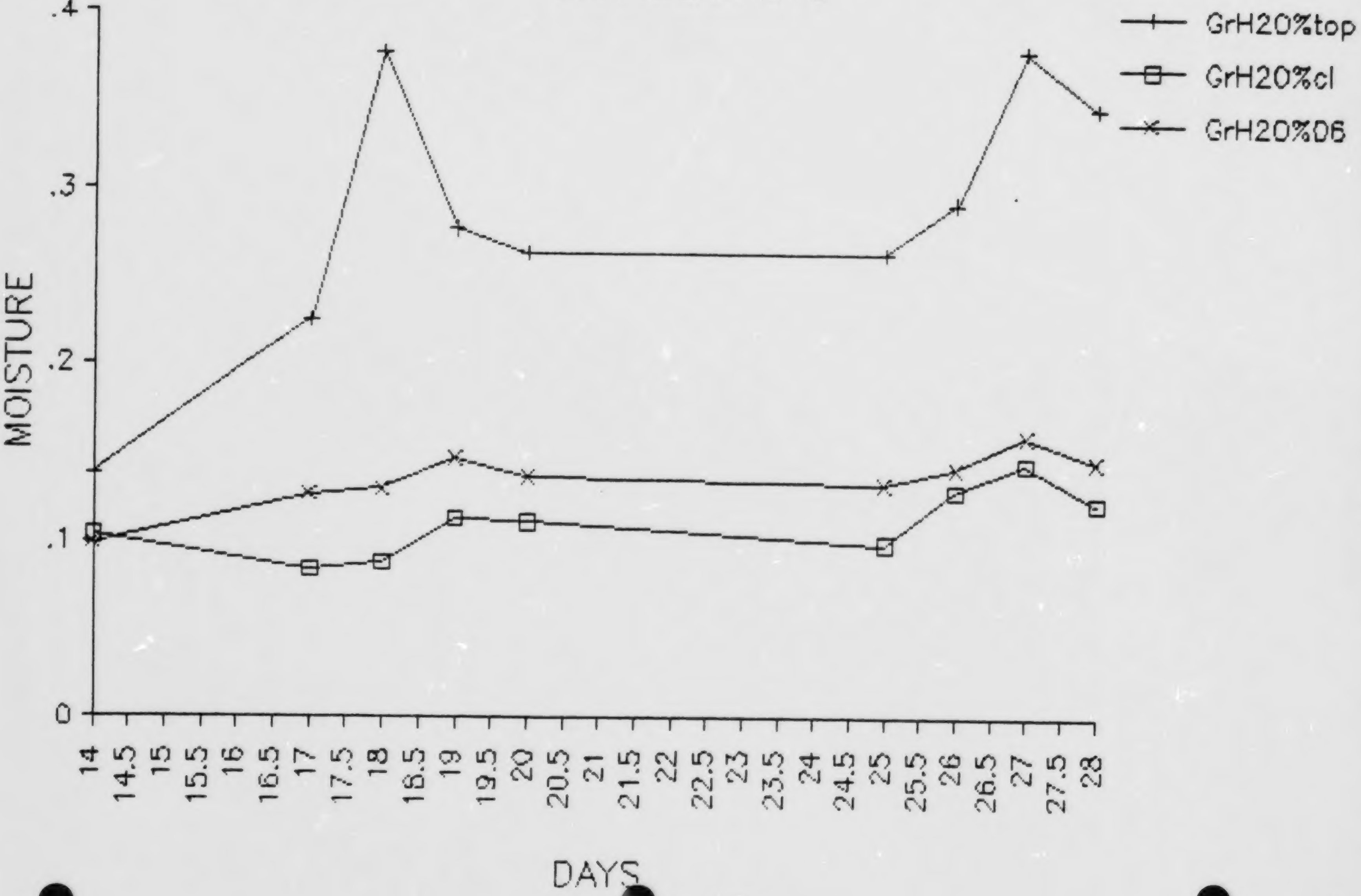
REL. HUM. (setting:70-75%)

GRANULAR 210



MOISTURE

GRANULAR 210



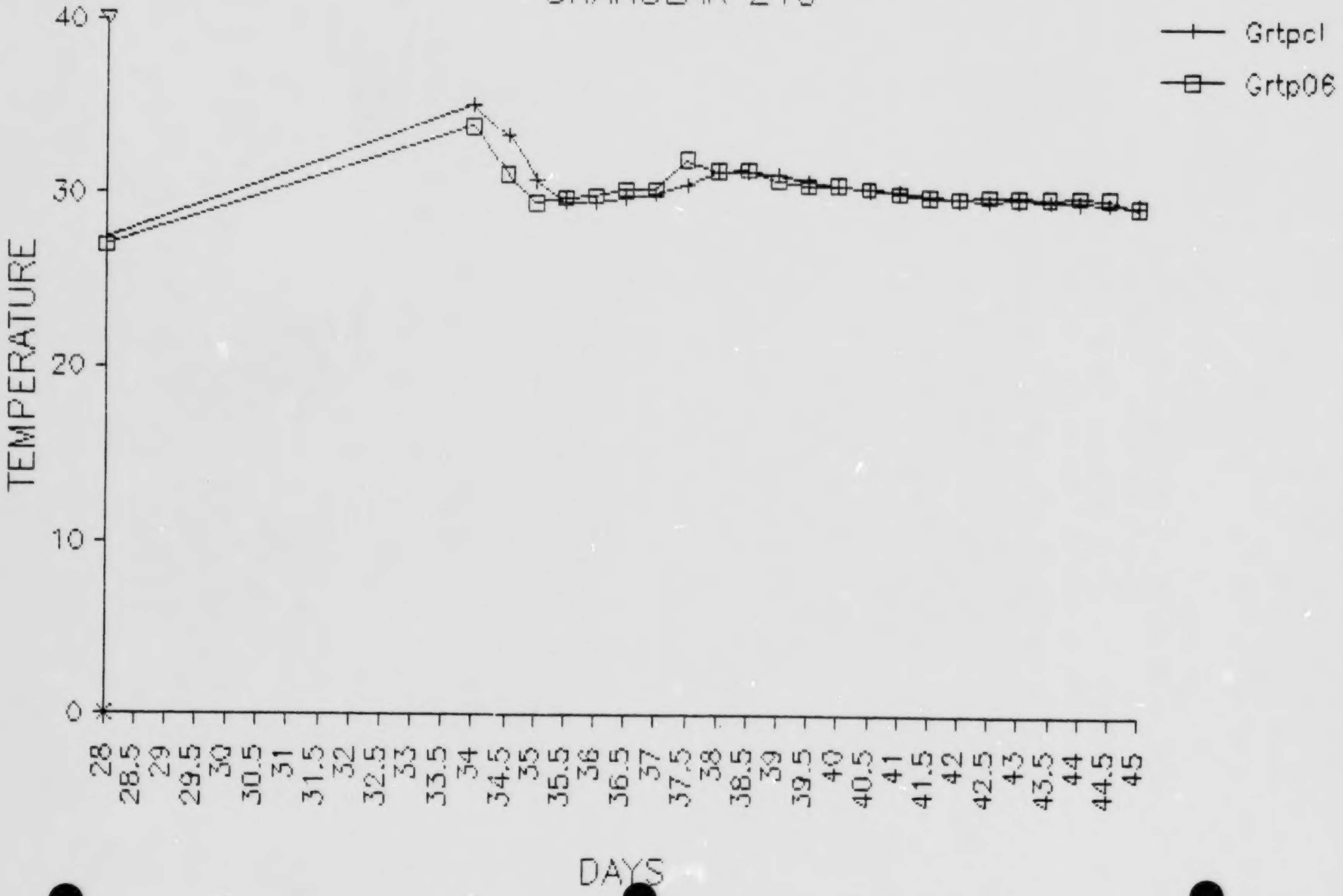
CLIMATE CHAMBER EXPERIMENT NO. 2

Setting Conditions: Temp. 25-30 C
R. H. 90-50% (1st cycle)

Date	Time	DAYS	Grrh%cl	Grrh%06	GrH20%cl	GrH20%06	GrH20%top	Grtpcl	Grtp06
05-27-88	12:48: 5	28	61.3	64.8	.122	.145	.347	27.341	26.92
05-28-88	0:01:00	28.5							
	12:01:00	29							
05-29-88	0:01:00	29.5							
	12:01:00	30							
05-30-88	0:01:00	30.5			.382	.236	5.221		
	12:01:00	31							
05-31-88	0:01:00	31.5			.552	.585	5.015		
	12:01:00	32							
06-01-88	0:01:00	32.5			.543	.635	5.605		
	12:01:00	33							
06-02-88	0:01:00	33.5			.944	1.21	5.394		
	12:24: 2	34	68.5	71.6				35.24	33.989
06-03-88	0:24: 3	34.5	69.1	71.6	.749	1.135	1.256	33.504	31.265
	12:48:56	35	69.5	71.2				30.879	29.614
06-04-88	0:48:57	35.5	69.9	71				29.71	29.919
	12:48:57	36	70.1	71				29.674	30.038
06-05-88	0:48:57	36.5	70.3	71				29.883	30.44
	12:48:58	37	70.7	70.8				30.138	30.44
06-06-88	0:48:58	37.5	70.7	70.8	1.125	.885	.238	30.668	32.152
	12:57:40	38	71.1	70.8				31.447	31.525
06-07-88	0:57:40	38.5	71.3	70.4	1.294	.976	.098	31.472	31.614
	12:57:41	39	71.3	69.8				31.311	30.945
06-08-88	0:57:41	39.5	71.5	69.4	.903	.763	.102	30.889	30.68
	12:57:42	40	71.5	69				30.666	30.729
06-09-88	0:57:42	40.5	71.7	68.8	.638	.683	.091	30.518	30.526
	12:57:43	41	71.9	68.4				30.41	30.301
06-10-88	0:57:43	41.5	72.1	68	.452	.454	.075	30.114	30.096
	12:30:34	42	72.1	67.2				29.982	30.017
06-11-88	0:30:35	42.5	72.3	66				29.881	30.185
	12:30:35	43	72.3	65				30.001	30.104
06-12-88	0:30:36	43.5	72.3	64.2				29.939	30.074
	12:30:36	44	72.3	63.6				29.843	30.134
06-13-88	0:30:37	44.5	72.5	63.2				29.791	30.074
	12:30:37	45	72.5	63	.445	.375	.085	29.711	29.569

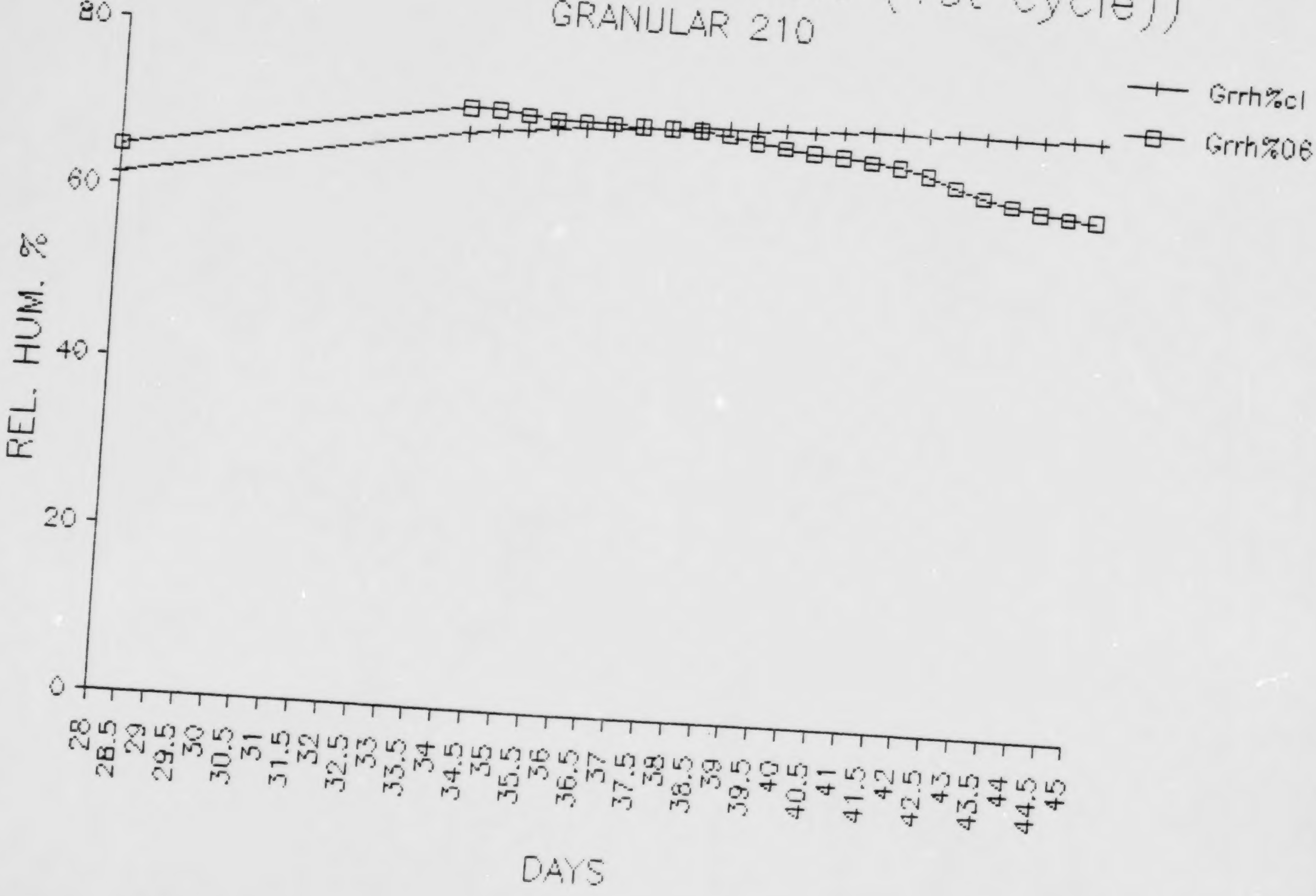
TEMPERATURE (setting:25-30 C)

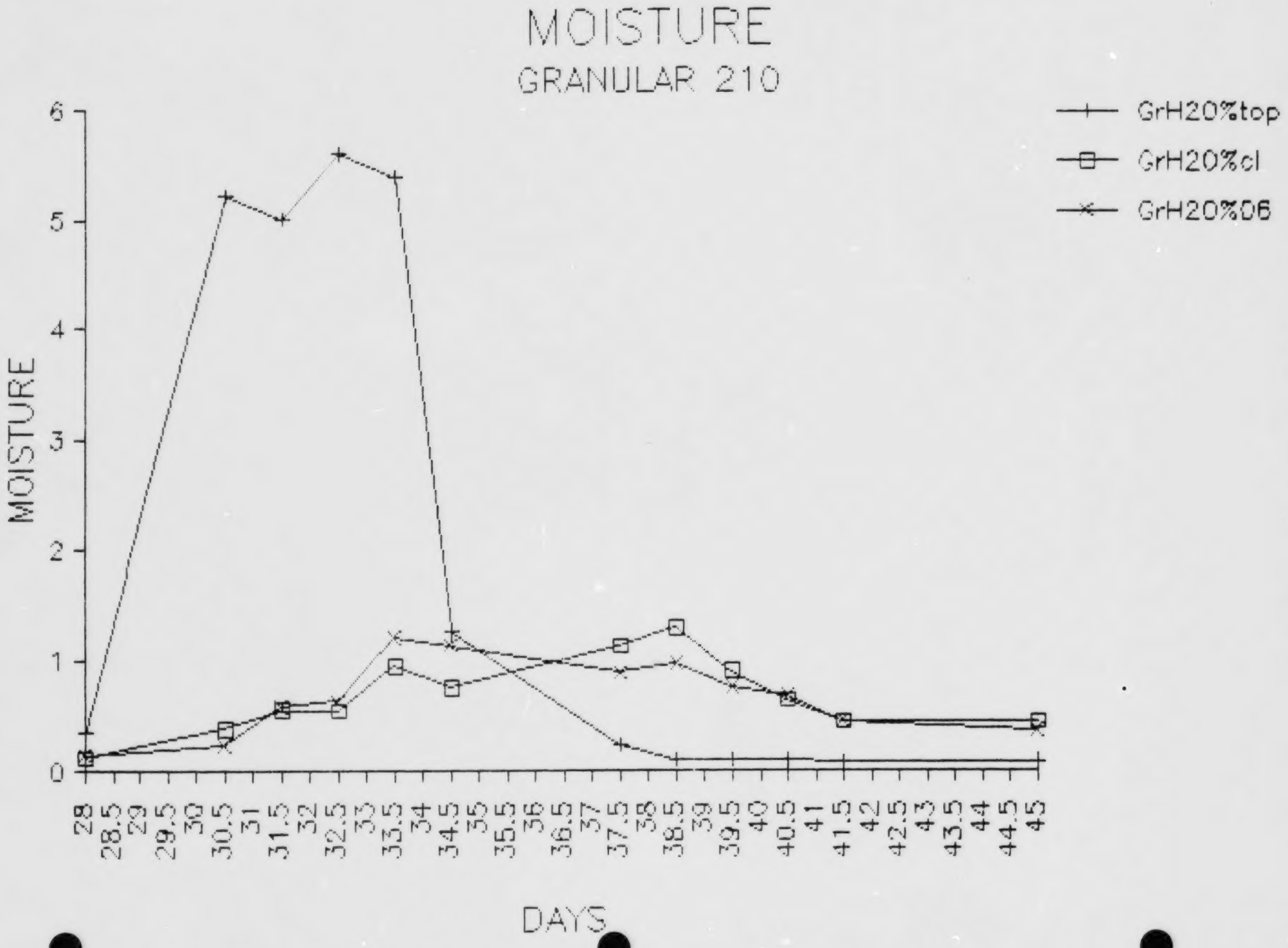
GRANULAR 210



REL.HUM.(setting:90–50% (1st cycle))

GRANULAR 210




MOISTURE
GRANULAR 210
GrH20%top
GrH20%cl
GrH20%06
MOISTURE
0
1
2
3
4
5
6
28
28.5
29
29.5
30
30.5
31
31.5
32
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37.5
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38.5
39
39.5
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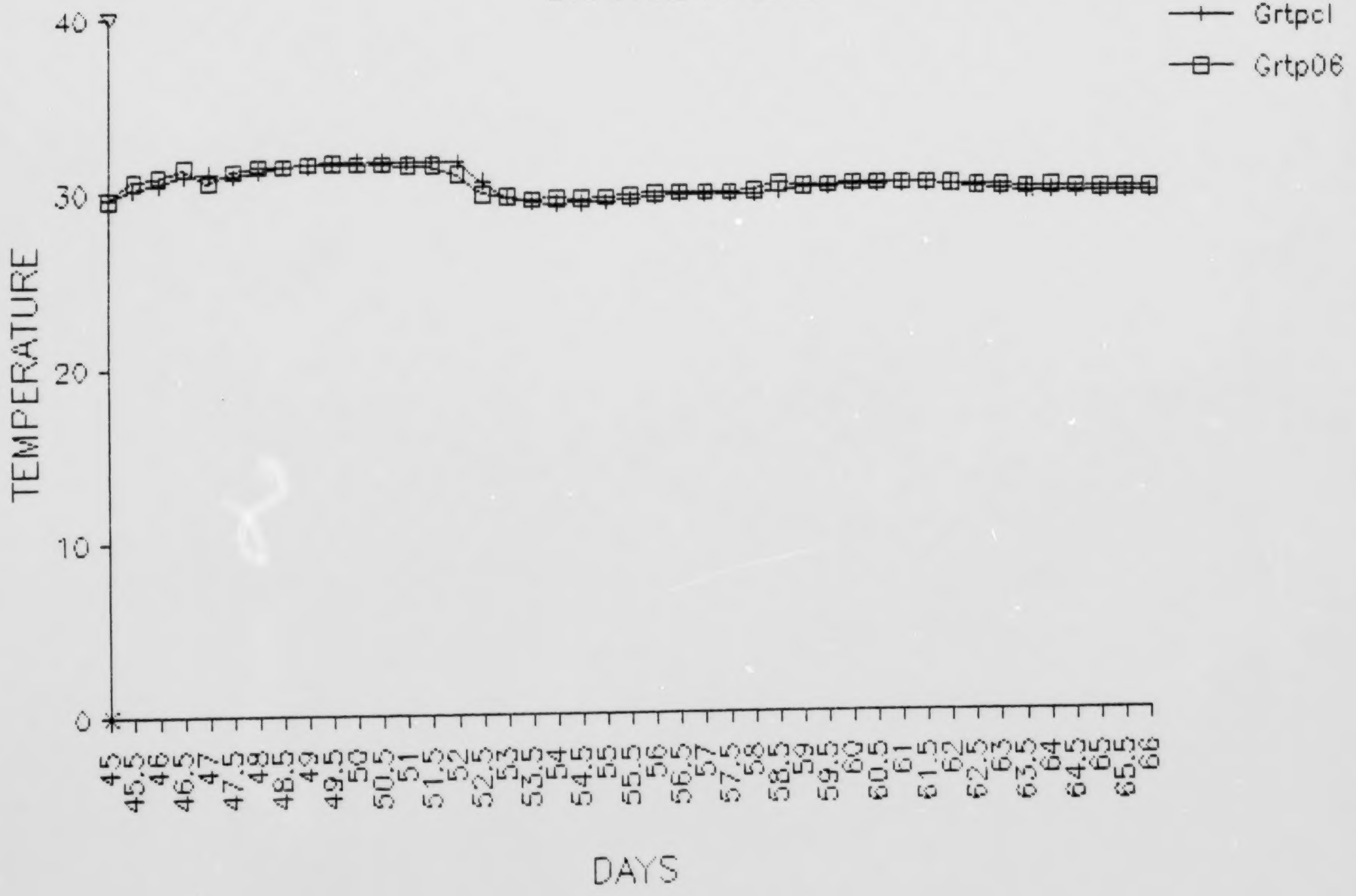
40.5
41
41.5
42
42.5
43
43.5
44
44.5
45
DAYS

TEMPERATURE (setting:25-30 C)

GRANULAR 210



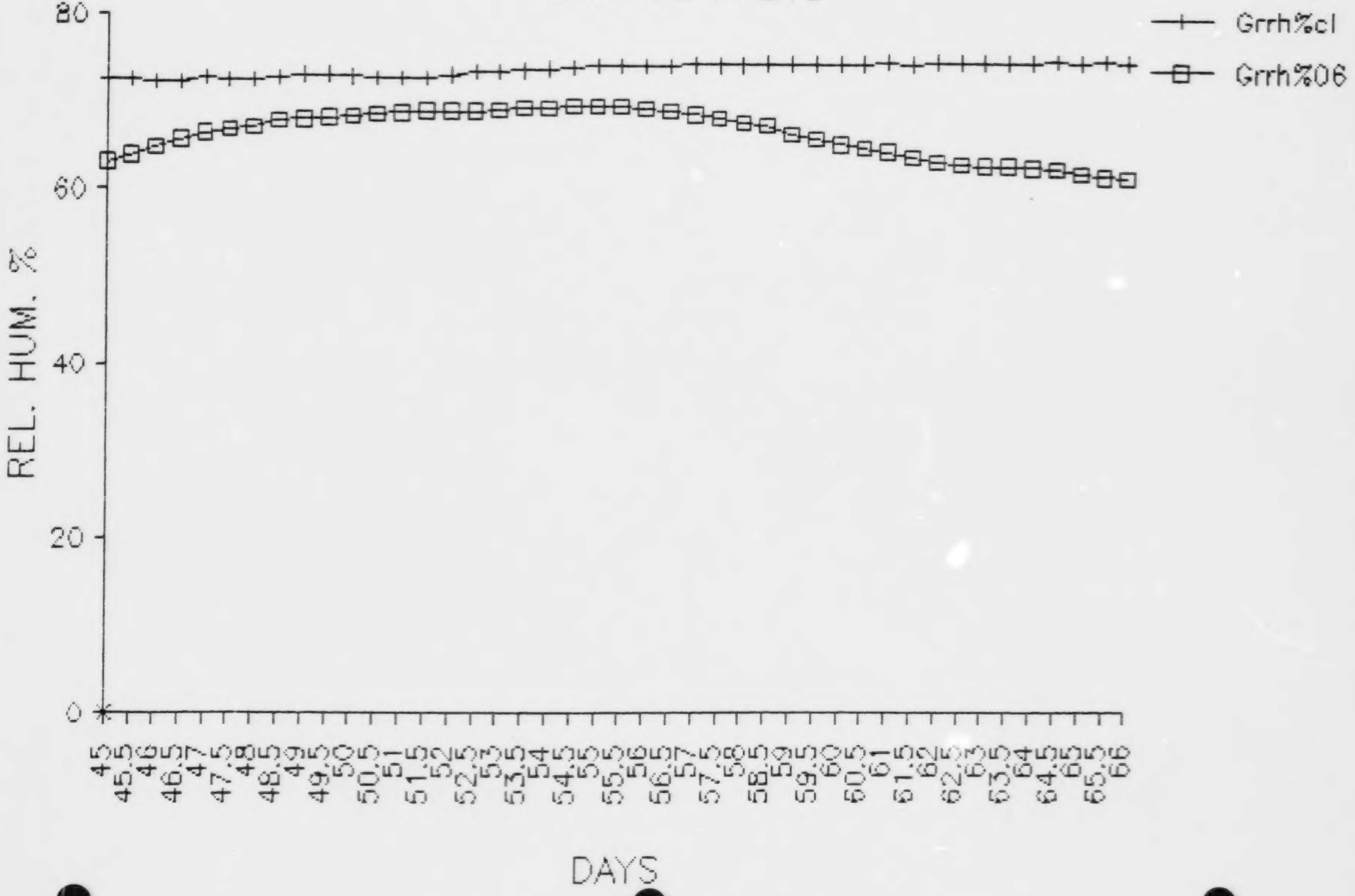
CLIMATE CHAMBER EXPERIMENT NO. 2

Setting Conditions: Temp. 25-30 C
R. H. 90-50% (2nd cycle)

Date	Time	DAYS	Grrh%cl	Grrh%06	GrH20%cl	GrH20%06	GrH20%top	Grtpcl	Grtp06
	12:30:37	45	72.5	63	.445	.375	.085	29.711	29.569
06-14-88	0:45:54	45.5	72.5	63.8				30.154	30.628
	12:45:55	46	72.3	64.8	.301	.299	3.933	30.518	30.855
06-15-88	0:45:55	46.5	72.3	65.6				30.913	31.393
	12:45:56	47	72.7	66.4	.297	.335	4.163	31.107	30.578
06-16-88	0:45:56	47.5	72.5	66.8				30.947	31.193
	12:00:00	48	72.5	67	.326	.272	4.521	31.249	31.461
06-17-88	0:45:57	48.5	72.7	67.8				31.465	31.485
	12:00:00	49	73.1	68	.376	.404	4.611	31.551	31.564
06-18-88	0: 0: 0	49.5	73.1	68.2				31.686	31.648
	12: 0: 0	50	72.9	68.4				31.76	31.567
06-19-88	0: 0: 1	50.5	72.7	68.6				31.757	31.596
	12: 0: 1	51	72.7	68.8				31.726	31.499
06-20-88	0: 0: 2	51.5	72.7	69				31.71	31.525
	12: 8:50	52	73.1	69	.996	.727	4.171	31.716	31
06-21-88	0: 8:50	52.5	73.5	69				30.592	29.853
	12: 8:51	53	73.5	69.2	.306	.33	.697	29.658	29.698
06-22-88	0: 8:51	53.5	73.7	69.4				29.363	29.514
	12: 8:51	54	73.9	69.4	.343	.374	.299	29.309	29.606
06-23-88	0: 8:52	54.5	74.1	69.6				29.331	29.578
	12: 8:52	55	74.3	69.6	.474	.443	.163	29.397	29.644
06-24-88	0: 8:53	55.5	74.3	69.6				29.508	29.726
	12: 8:54	56	74.3	69.4	.403	.387	.088	29.674	29.865
06-25-88	0: 8:54	56.5	74.3	69.2				29.777	29.881
	12: 8:55	57	74.5	68.8				29.772	29.935
06-26-88	0: 8:55	57.5	74.5	68.4				29.761	29.929
	12: 8:56	58	74.5	67.8				29.777	29.982
06-27-88	0: 8:56	58.5	74.7	67.6				29.907	30.382
	12:11:24	59	74.7	66.6	.3	.358	.072	30.158	30.259
06-28-88	0:11:24	59.5	74.7	66				30.208	30.304
	12:11:25	60	74.7	65.4	.372	.36	.059	30.272	30.408
06-29-88	0:11:25	60.5	74.7	65				30.344	30.422
	12:11:26	61	74.9	64.6	.374	.297	.065	30.372	30.4
06-30-88	0:11:27	61.5	74.7	64				30.372	30.444
	12: 6:51	62	74.9	63.4				30.336	30.356
07-01-88	0: 6:51	62.5	74.9	63.2				30.168	30.242
	12: 6:52	63	74.9	63				30.058	30.258
07-02-88	0: 6:53	63.5	74.9	63				29.972	30.15
	12: 6:53	64	74.9	62.8				29.947	30.201
07-03-88	0: 6:54	64.5	75.1	62.6				29.931	30.138
	12: 6:54	65	74.9	62.2				29.917	30.13
07-04-88	0: 6:55	65.5	75.1	61.8				29.923	30.124
	8: 6:55	66	74.9	61.6	.316	.185	.074	29.923	30.122

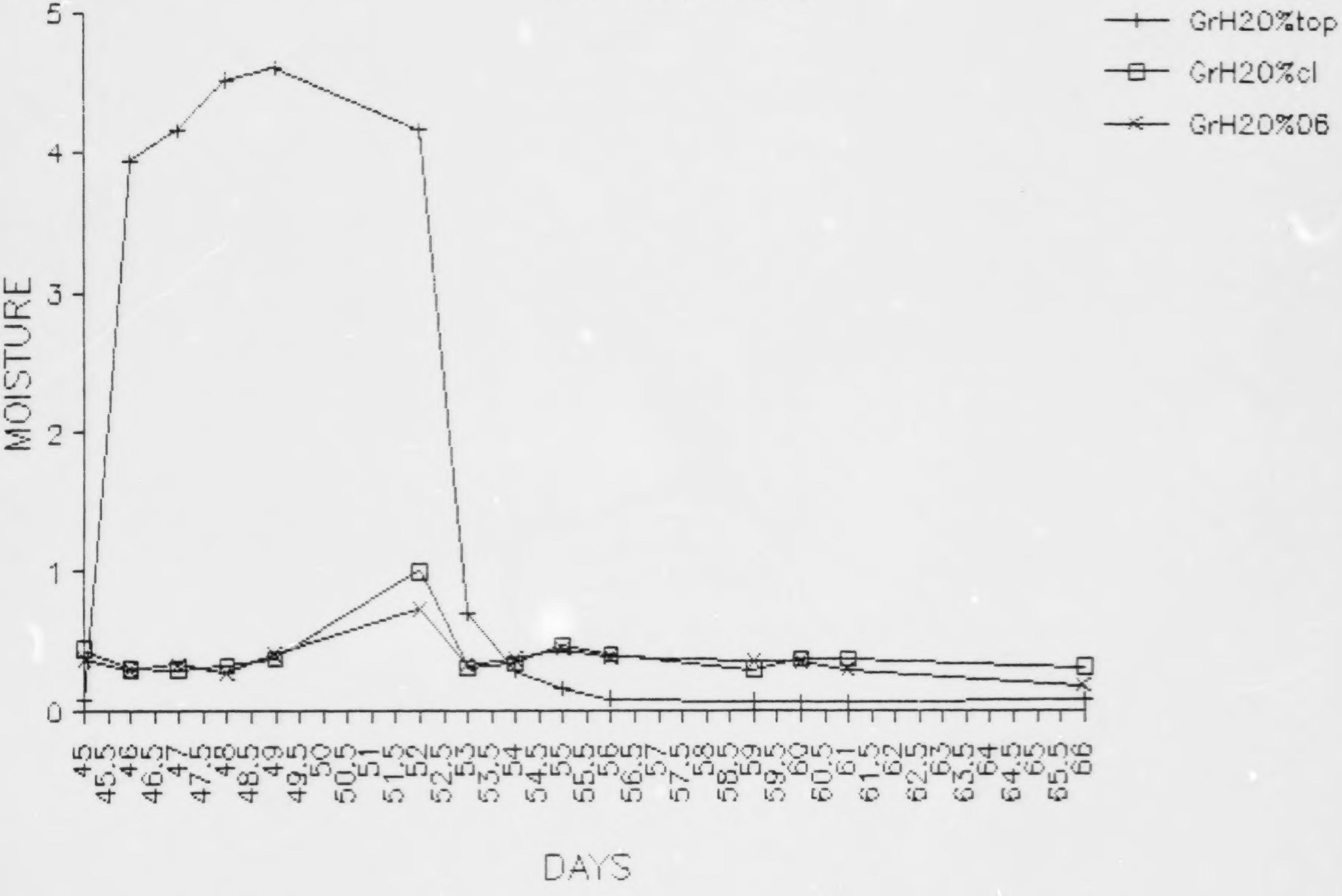
REL.HUM.(setting:90–50% (2nd cycle))

GRANULAR 210



MOISTURE

GRANULAR 210



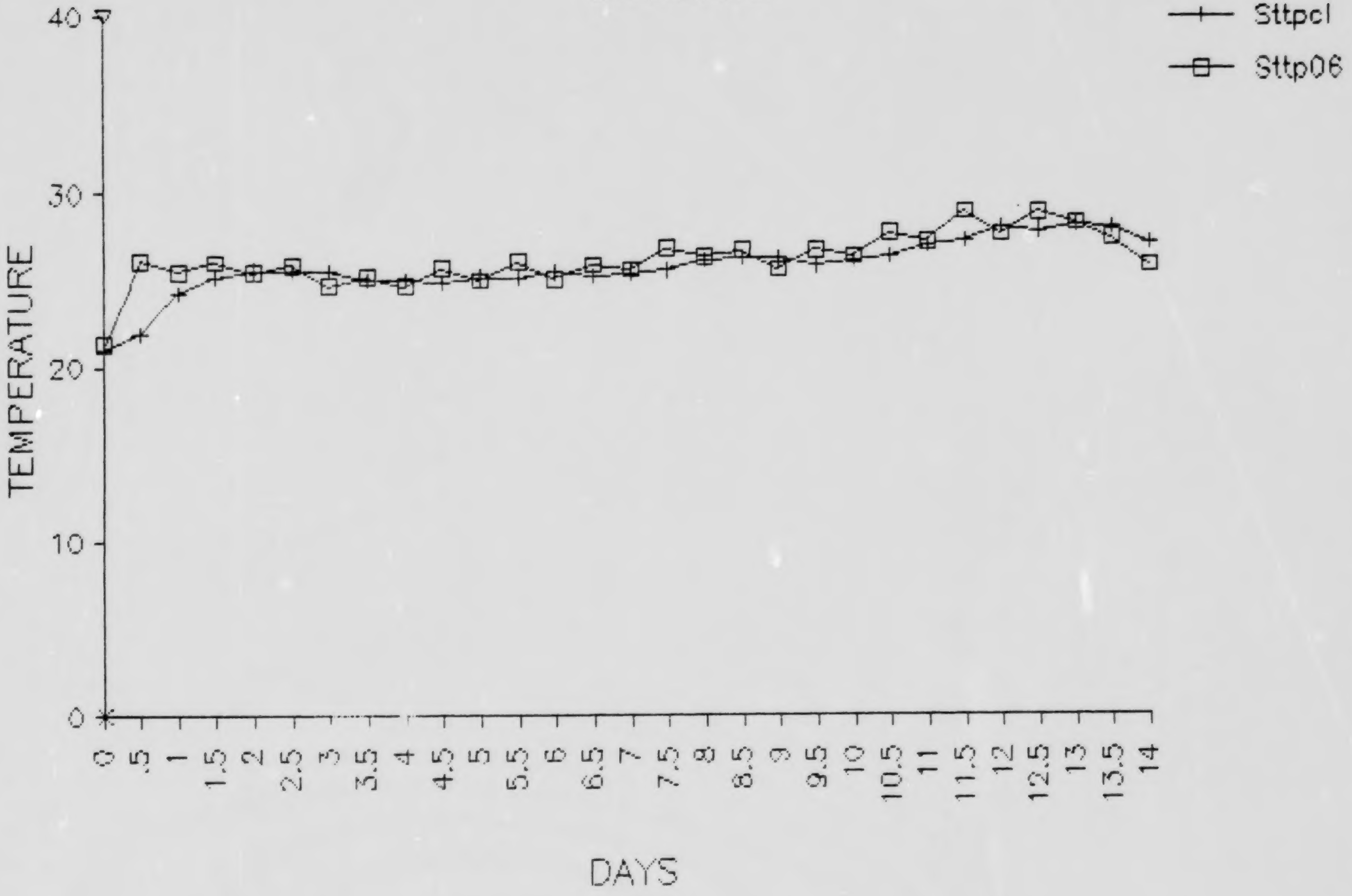
CLIMATE CHAMBER EXPERIMENT NO. 2

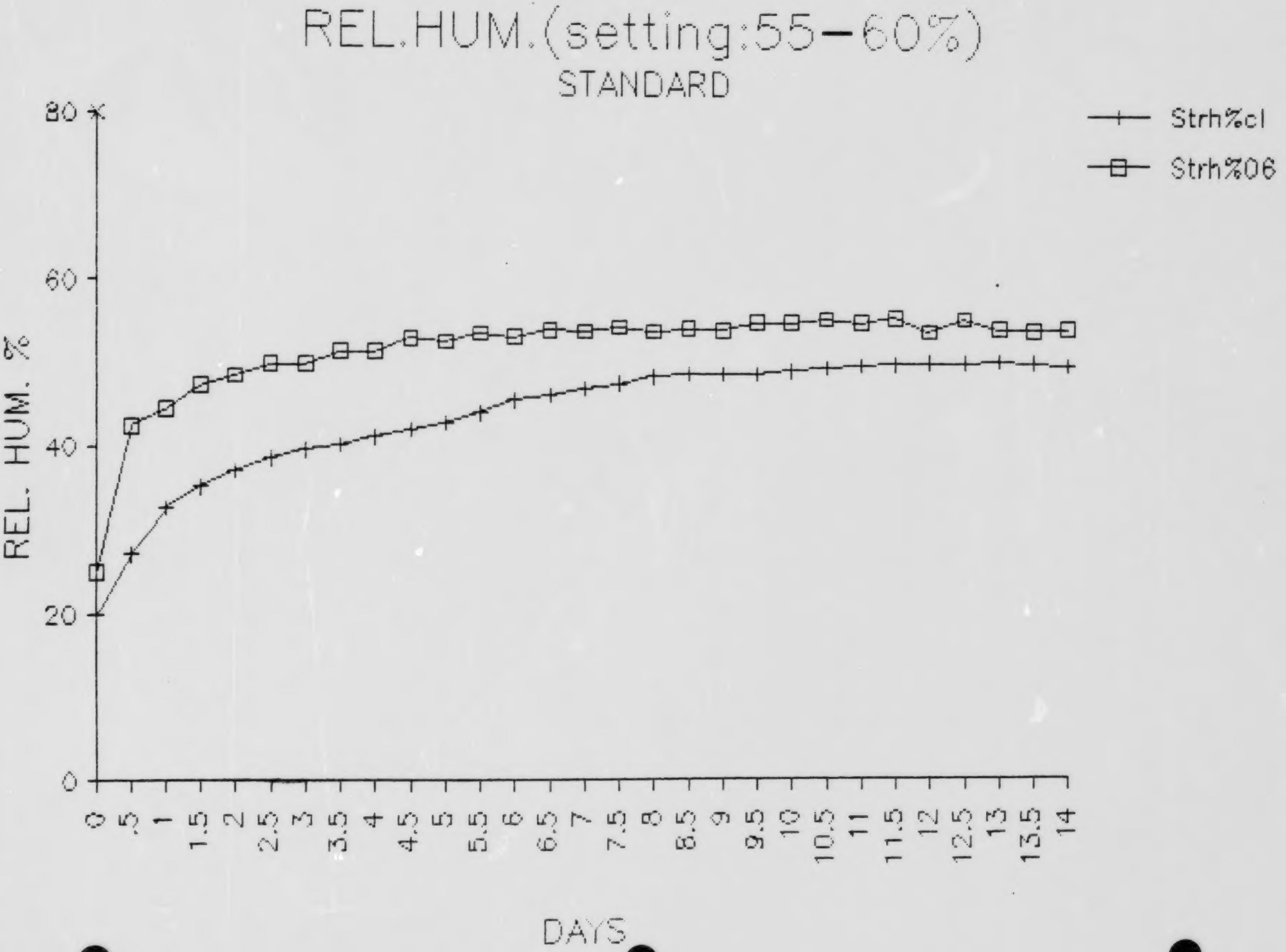
Setting Conditions: Temp. 25-30 C
R. H. 55-60%

Date	Time	DAYS	Strh%cl	Strh%06	StH20%cl	StH20%06	StH20%top	Sttpcl	Sttp06
04-29-88	11:34:24	0	19.9	25	.059	.059	.059	21.024	21.269
04-30-88	0: 2:38	.5	27.1	42.4				21.887	26.091
	12: 2:38	1	32.7	44.4				24.235	25.447
05-01-88	0: 2:39	1.5	35.1	47.2				25.158	26.031
	12: 2:39	2	37.1	48.4				25.567	25.426
05-02-88	0: 2:40	2.5	38.5	49.8				25.511	25.846
	12: 2:40	3	39.5	49.8	.056	.09	.107	25.517	24.65
05-03-88	0: 2:40	3.5	40.1	51.4				25.019	25.182
	12: 2:41	4	41.1	51.4	.049	.083	.107	24.958	24.66
05-04-88	0: 2:42	4.5	41.9	53				24.839	25.691
	12: 2:42	5	42.7	52.6	.06	.088	.117	25.152	24.99
05-05-88	0: 2:43	5.5	43.9	53.6				25.132	26.073
	12: 2:43	6	45.5	53.2	.065	.1	.144	25.461	25.056
05-06-88	0: 2:43	6.5	46.1	54				25.219	25.903
	12:51:48	7	46.9	53.8	.063	.08	.109	25.439	25.638
05-07-88	0:51:48	7.5	47.5	54.4				25.586	26.861
	12:59:58	8	48.3	53.8				26.174	26.374
05-08-88	0:59:58	8.5	48.7	54.2				26.288	26.712
	12:59:59	9	48.7	54				26.221	25.606
05-09-88	0:59:59	9.5	48.7	55				25.846	26.761
	12: 0: 0	10	49.1	55				26.128	26.423
05-10-88	0: 0: 0	10.5	49.5	55.4				26.392	27.743
	12: 0: 1	11	49.7	55				27.058	27.266
05-11-88	0: 0: 1	11.5	49.9	55.6				27.337	28.993
	12: 0: 1	12	50.1	53.8	.073	.103	.124	28.108	27.687
05-12-88	0: 0: 2	12.5	50.1	55.4				27.871	28.945
	12: 0: 3	13	50.3	54.2	.069	.103	.12	28.285	28.358
05-13-88	0: 0: 3	13.5	50.1	54				28.077	27.51
	12:47: 3	14	49.7	54.2	.086	.109	.108	27.215	25.893

TEMPERATURE (setting:25-30 C)

STANDARD




REL.HUM.(setting:55—60%)
STANDARD
Strh%cl
Strh%06
REL. HUM. %
80
60
40
20
0
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.5
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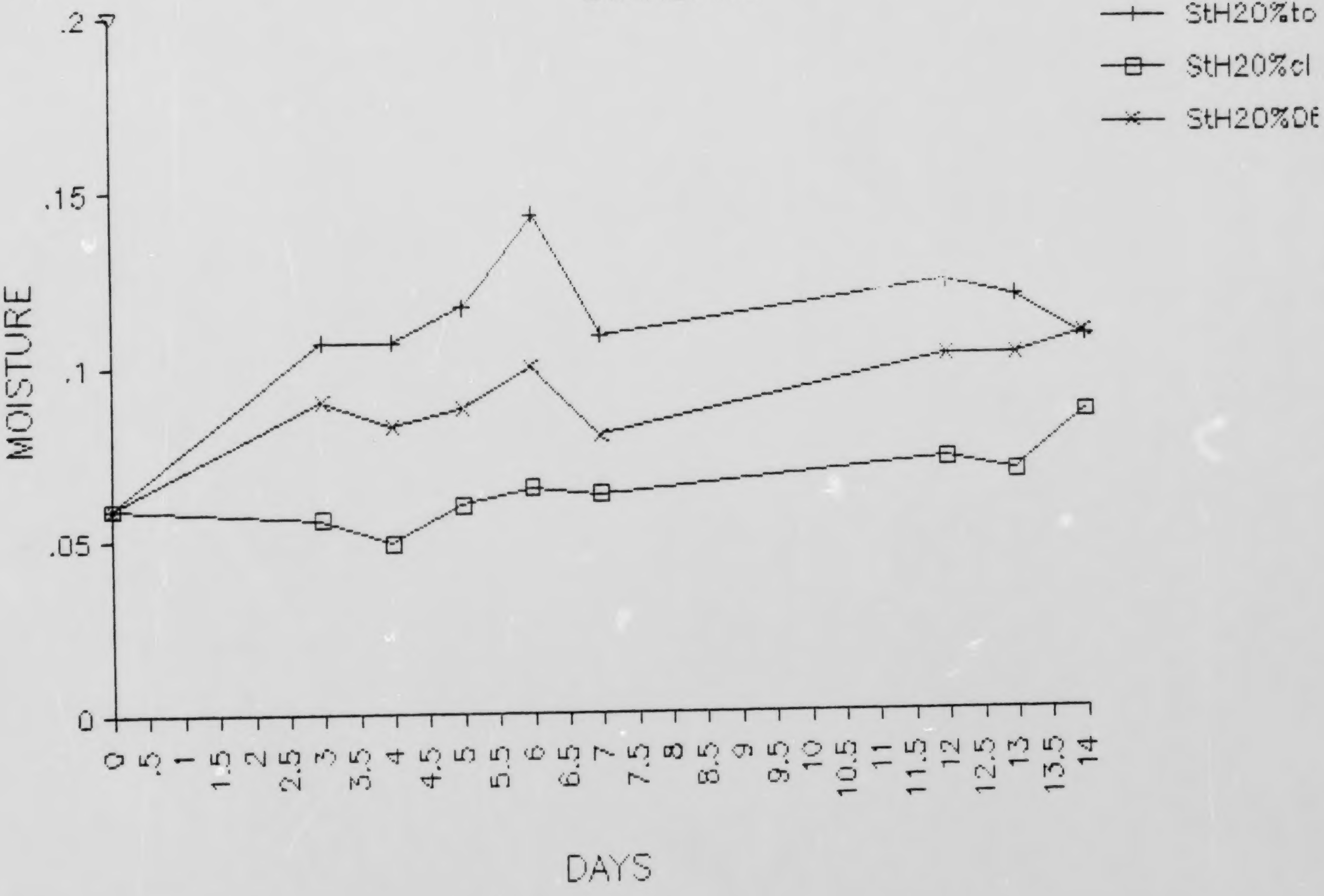
14
DAYS

MOISTURE

STANDARD

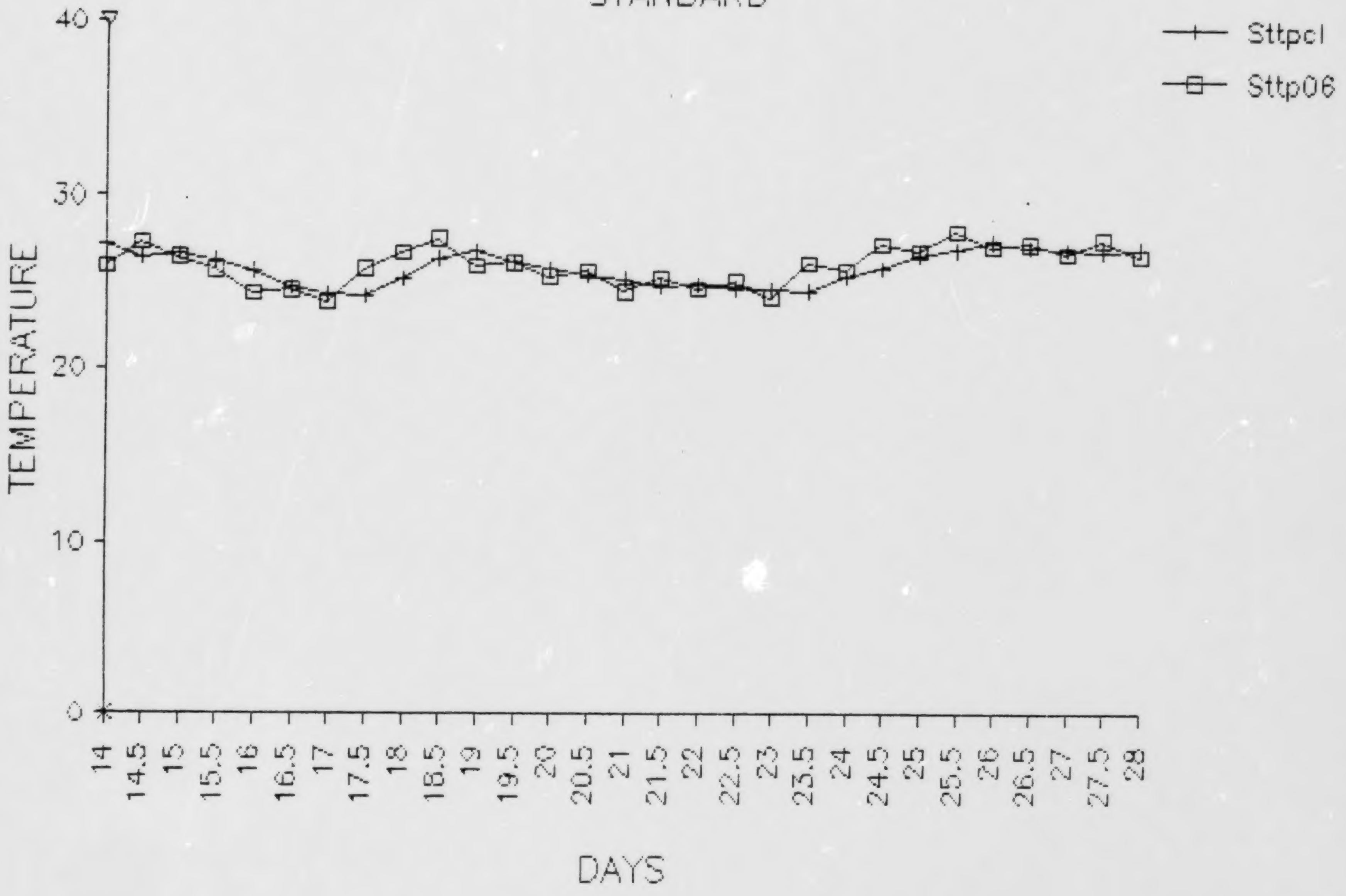


CLIMATE CHAMBER EXPERIMENT NO. 2

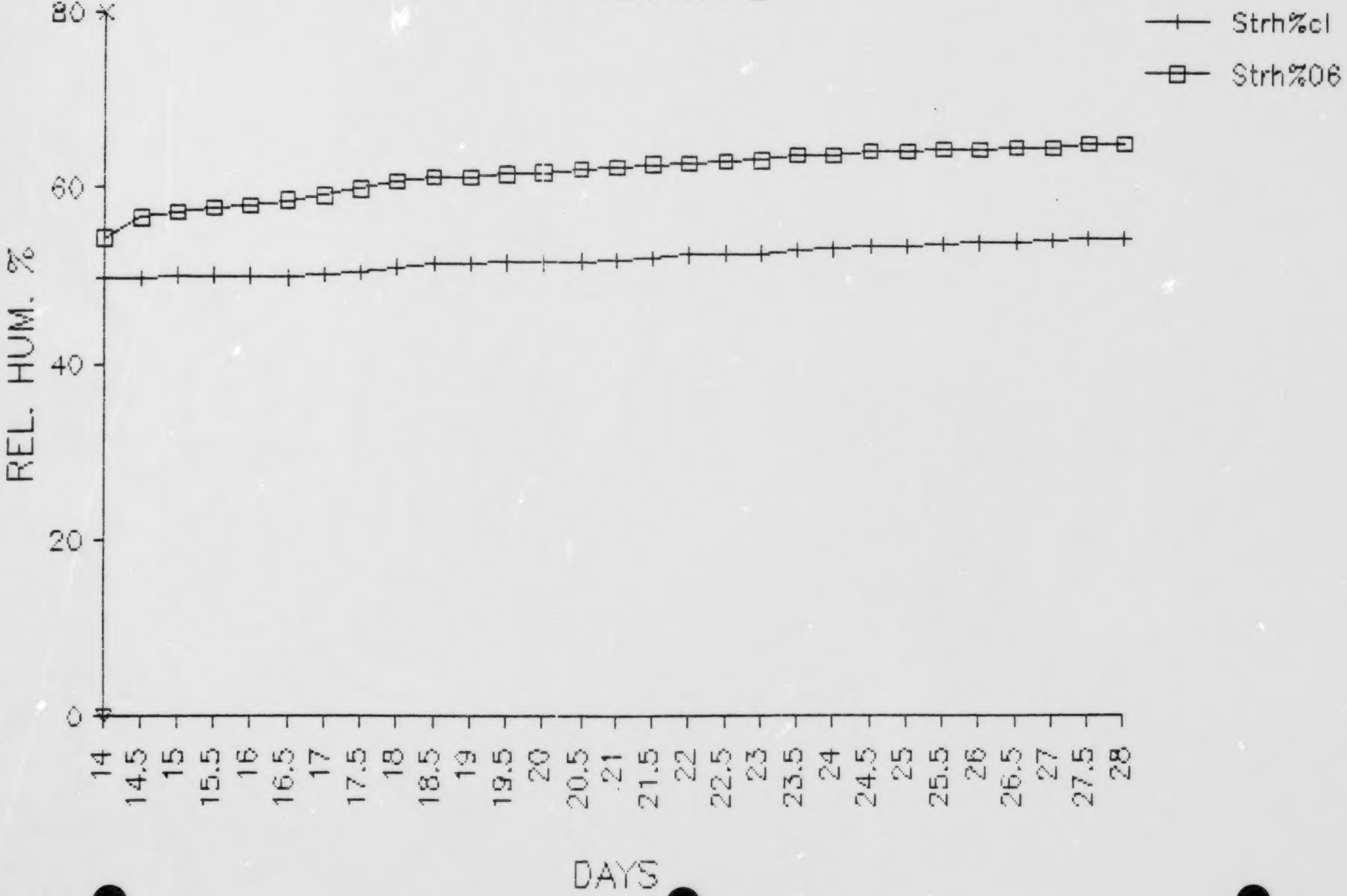
Setting Conditions: Temp. 25-30 C
R. H. 70%

Date	Time	DAYS	Strh%cl	Strh%06	StH20%cl	StH20%06	StH20%top	Sttpcl	Sttp06
05-13-88	12:47: 3	14	49.7	54.2	.086	.109	.108	27.215	25.893
05-14-88	0:47: 4	14.5	49.7	56.6				26.445	27.345
	12:47: 4	15	50.1	57.2				26.684	26.451
05-15-88	0:47: 5	15.5	50.1	57.8				26.329	25.73
	12:47: 5	16	50.1	58				25.67	24.318
05-16-88	0:47: 6	16.5	49.9	58.6				24.736	24.546
	12:47: 6	17	50.3	59.2	.081	.106	.191	24.291	23.85
05-17-88	0:47: 7	17.5	50.5	60				24.217	25.817
	12:47: 7	18	51.1	60.8	.073	.106	.33	25.266	26.787
05-18-88	0:47: 8	18.5	51.5	61.4				26.463	27.638
	12:47: 8	19	51.5	61.4	.087	.14	.25	26.891	26.028
05-19-88	0:47: 9	19.5	51.7	61.8				26.235	26.235
	12:48:58	20	51.7	62	.104	.14	.226	25.901	25.412
05-20-88	0:48:59	20.5	51.9	62.4				25.517	25.758
	12:48:59	21	52.1	62.6	.063	.08	.109	25.292	24.542
05-21-88	0:49: 0	21.5	52.3	63				24.831	25.398
	12:49: 0	22	52.7	63.2				25.013	24.746
05-22-88	0:49: 0	22.5	52.9	63.4				24.806	25.174
	12:49: 1	23	52.9	63.6				24.681	24.266
05-23-88	0:49: 1	23.5	53.3	64.2				24.662	26.264
	12:49: 2	24	53.5	64.2				25.539	25.917
05-24-88	0:49: 2	24.5	53.9	64.8				26.054	27.426
	12:49: 3	25	53.9	64.8	.1	.127	.208	26.84	27.072
05-25-88	0:49: 3	25.5	54.1	65				27.135	28.208
	12:48: 4	26	54.3	65	.147	.142	.349	27.632	27.304
05-26-88	0:48: 4	26.5	54.5	65.2				27.293	27.529
	12:48: 5	27	54.7	65.2	.145	.171	.279	27.16	26.948
05-27-88	0:48: 5	27.5	54.9	65.8				27.123	27.749
	10:48: 5	28	54.9	65.8	.101	.133	.274	27.246	26.819

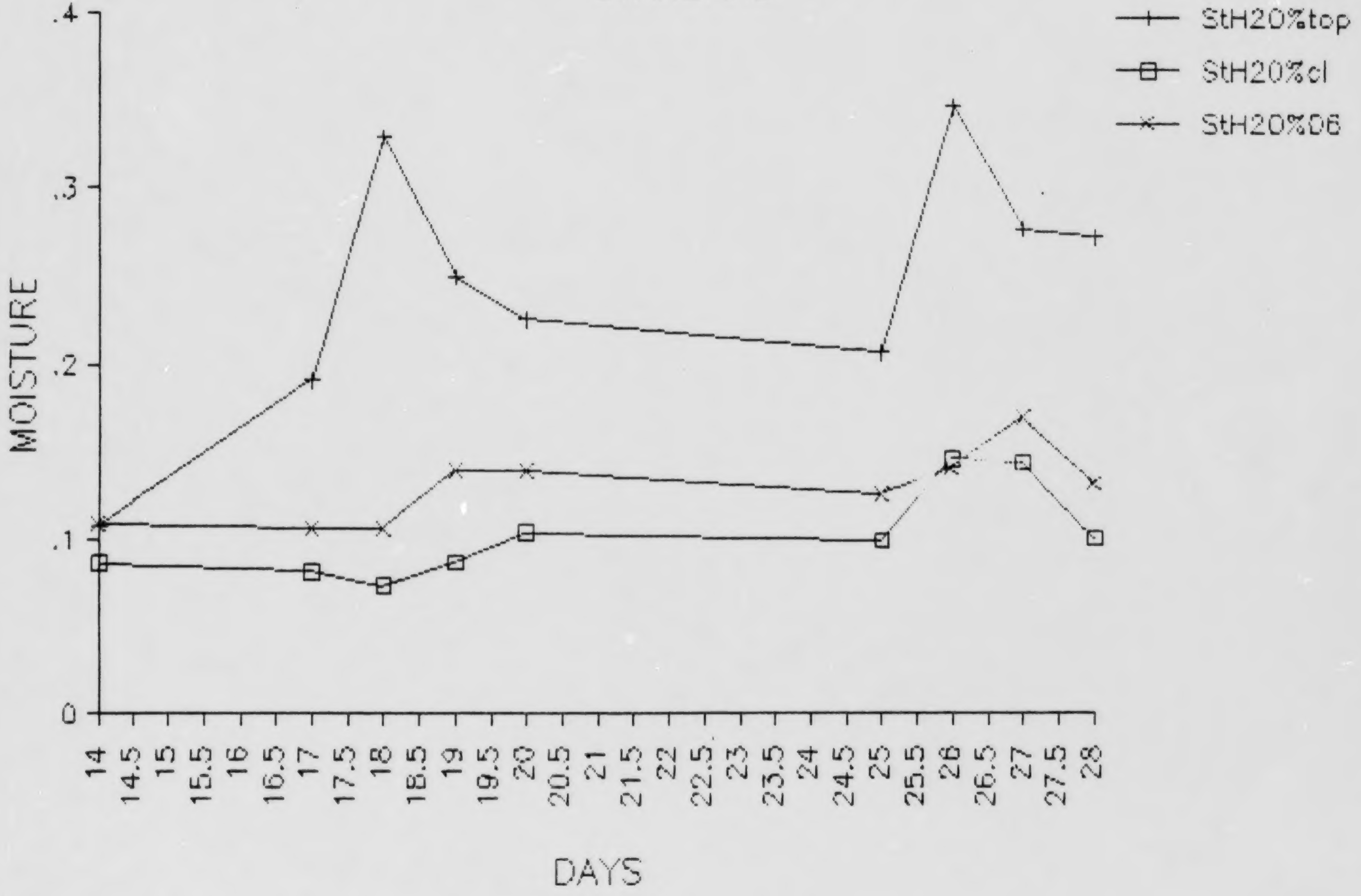
TEMPERATURE (setting:25-30 C)
STANDARD



REL. HUM. (setting:70-75%)
STANDARD



MOISTURE
STANDARD



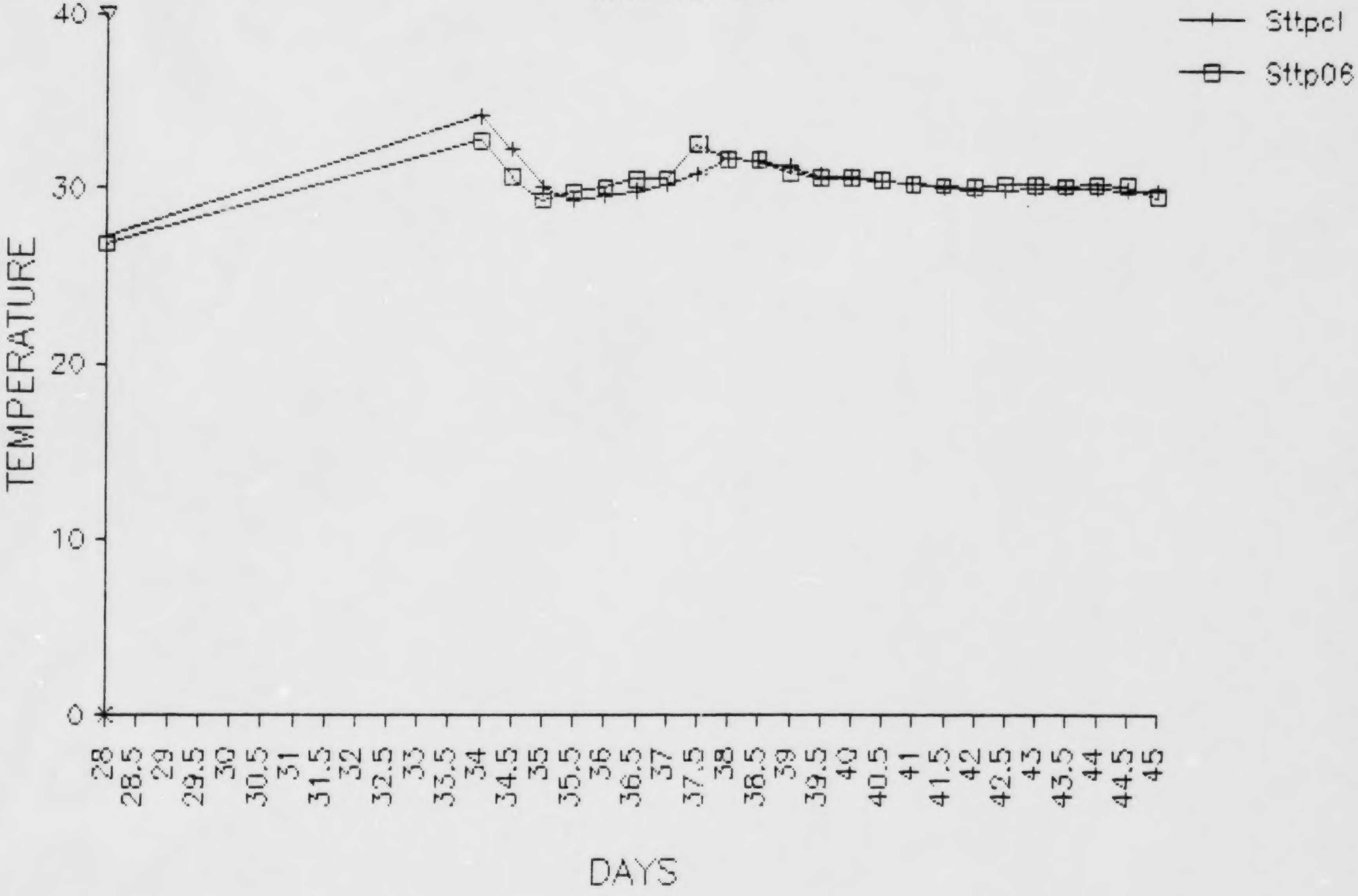
CLIMATE CHAMBER EXPERIMENT NO. 2

Setting Conditions: Temp. 25-30 C:
R. H. 90-50%

Date	Time	DAYS	Strh%cl	Strh%06	StH20%cl	StH20%06	StH20%top	Sttpcl	Sttp06
05-27-88	12:48: 5	28	54.9	65.8	.101	.133	.274	27.246	26.819
05-28-88	0:01:00	28.5							
	12:01:00	29							
05-29-88	0:01:00	29.5							
	12:01:00	30							
05-30-88	0:01:00	30.5			.523	.389	5.539		
	12:01:00	31							
05-31-88	0:01:00	31.5			.536	.925	5.87		
	12:01:00	32							
06-01-88	0:01:00	32.5			1.697	.951	5.528		
	12:01:00	33							
06-02-88	0:01:00	33.5			1.503	1.35	8.364		
	12:24: 2	34	68.9	72.6				34.259	32.823
06-03-88	0:24: 3	34.5	69.1	72.8	3.122	2.22	.709	32.378	30.747
	12:48:56	35	69.3	73				30.12	29.367
06-04-88	0:48:57	35.5	69.5	73				29.385	29.931
	12:48:57	36	69.5	73.2				29.62	30.162
06-05-88	0:48:57	36.5	69.5	73.4				29.972	30.65
	12:48:58	37	69.7	73.4				30.278	30.682
06-06-88	0:48:58	37.5	69.7	73.4	1.781	.931	.205	30.968	32.674
	12:57:40	38	69.7	73.6				31.827	31.809
06-07-88	0:57:40	38.5	69.9	73.6	1.722	1.223	.164	31.694	31.773
	12:57:41	39	69.9	73.6				31.381	31.013
06-08-88	0:57:41	39.5	69.9	73.6	.286	.6	.141	30.871	30.729
	12:57:42	40	69.9	73.4				30.652	30.763
06-09-88	0:57:42	40.5	69.9	73.4	.599	.426	.192	30.534	30.602
	12:57:43	41	69.9	73.2				30.43	30.388
06-10-88	0:57:43	41.5	69.9	73	.402	.377	.105	30.172	30.266
	12:30:34	42	69.9	72.8				30.072	30.222
06-11-88	0:30:35	42.5	69.7	72.2				30.068	30.412
	12:30:35	43	69.7	71.4				30.228	30.376
06-12-88	0:30:36	43.5	69.7	70.6				30.17	30.32
	12:30:36	44	69.5	70				30.104	30.384
06-13-88	0:30:37	44.5	69.5	69.4				30.09	30.334
	12:30:37	45	69.5	69	.534	.319	.102	29.988	29.694

TEMPERATURE (setting:25-30 C)

STANDARD



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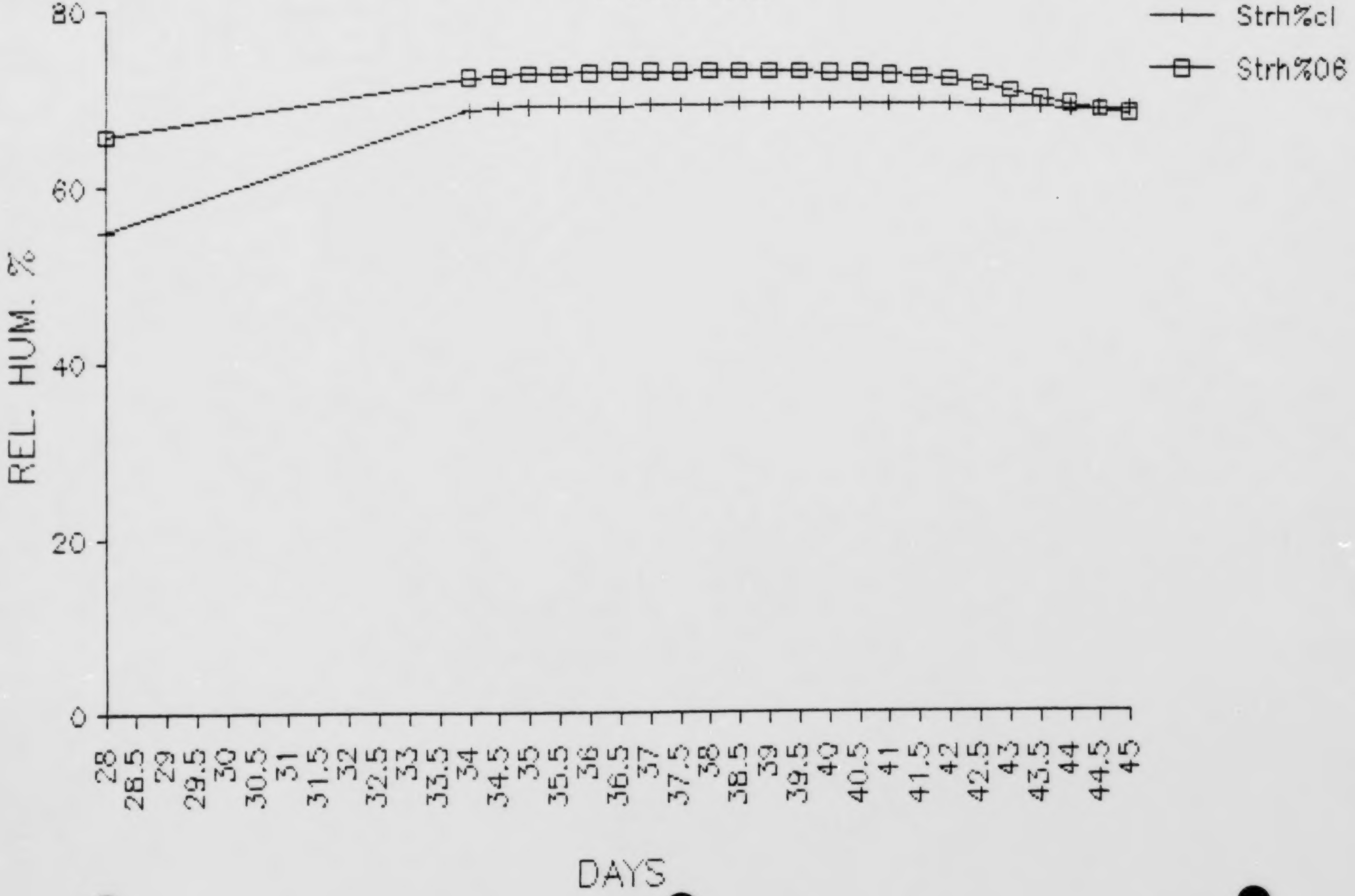
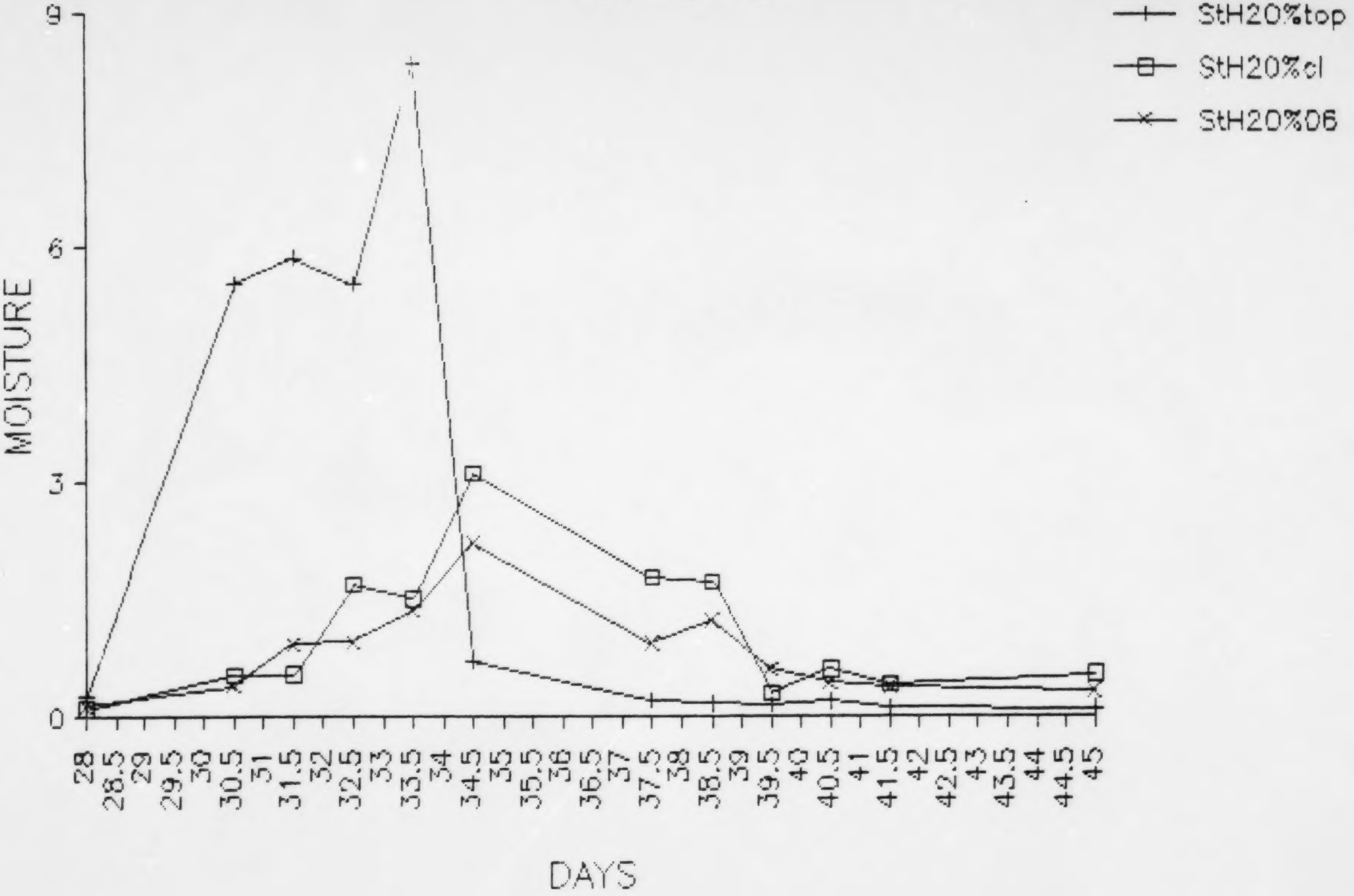


MOISTURE
STANDARD



CLIMATE CHAMBER EXPERIMENT NO. 2

Setting Conditions: Temp. 25-30 C
R. H. 90-50% (2nd cycle)

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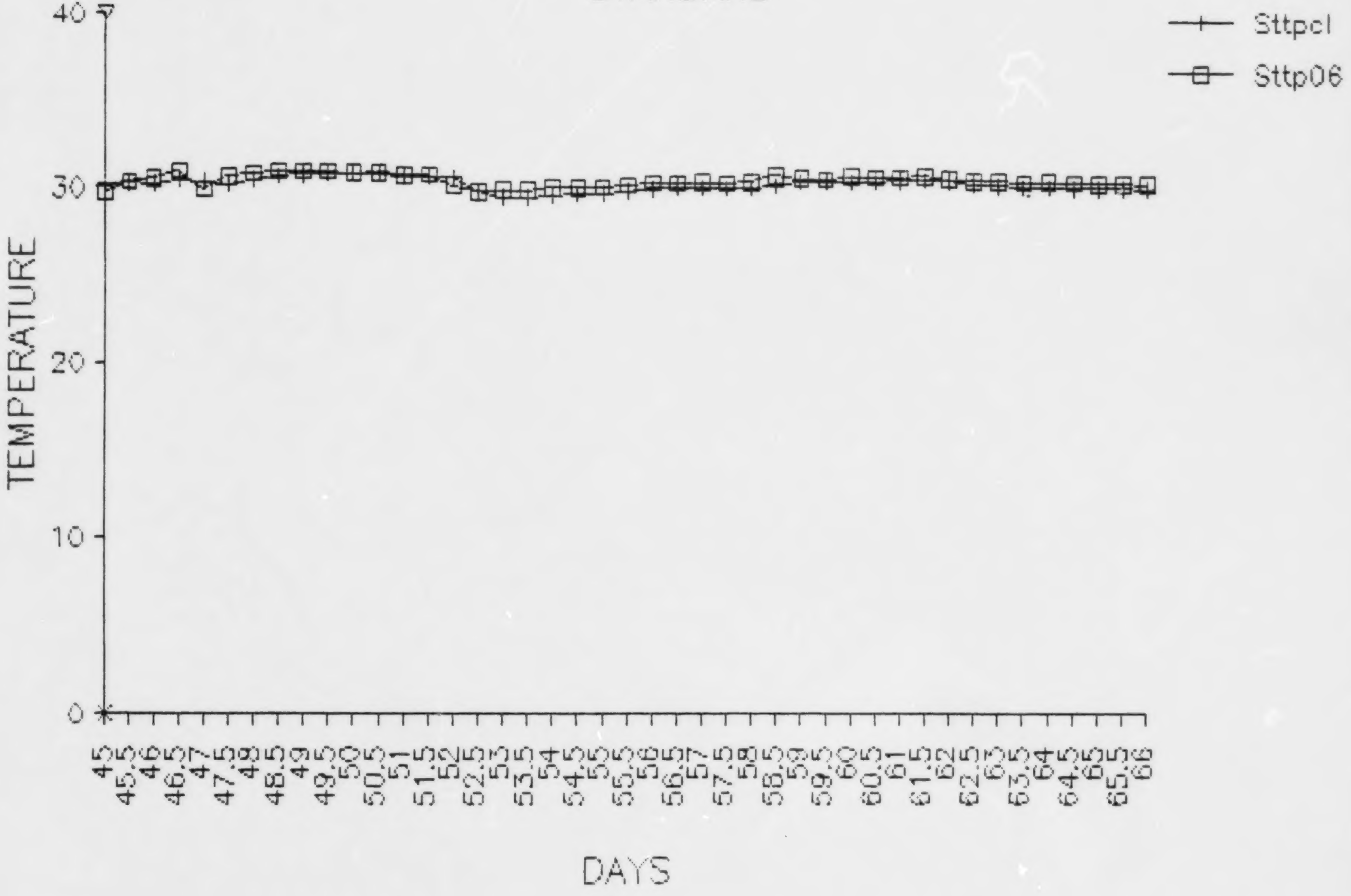
	12:30:37	45	69.5	69	.534	.319	.102	29.988	29.694
06-14-88	0:45:54	45.5	69.5	69.4				30.246	30.294
	12:45:55	46	69.5	70.2	.378	.403	4.267	30.201	30.516
06-15-88	0:45:55	46.5	69.5	70.6				30.438	30.921
	12:45:56	47	69.5	70.8	.409	.43	4.489	30.33	29.919
06-16-88	0:45:56	47.5	69.3	71				30.14	30.648
	12:00:00	48	69.5	71.2	.457	.604	4.214	30.486	30.823
06-17-88	0:45:57	48.5	69.5	71.4				30.694	30.939
	12:00:00	49	69.5	71.4	.471	.673	5.899	30.752	30.945
06-18-88	0: 0: 0	49.5	69.7	71.6				30.837	30.95
	12: 0: 0	50	69.5	71.6				30.826	30.863
06-19-88	0: 0: 1	50.5	69.5	71.8				30.798	30.892
	12: 0: 1	51	69.5	71.8				30.748	30.779
06-20-88	0: 0: 2	51.5	69.9	71.8				30.7	30.787
	12: 8:50	52	69.9	71.8	.687	.726	4.541	30.684	30.2
06-21-88	0: 8:50	52.5	70.1	72				29.917	29.873
	12: 8:51	53	70.1	72	.454	.482	.22	29.562	29.963
06-22-88	0: 8:51	53.5	70.3	72.2				29.57	29.969
	12: 8:51	54	70.3	72.2	.326	.426	.155	29.656	30.132
06-23-88	0: 8:52	54.5	70.3	72.2				29.745	30.124
	12: 8:52	55	70.3	72	.414	.406	.11	29.825	30.19
06-24-88	0: 8:53	55.5	70.3	71.8				29.947	30.278
	12: 8:54	56	70.3	71.2	.359	.41	.095	30.102	30.436
06-25-88	0: 8:54	56.5	70.3	70.6				30.201	30.442
	12: 8:55	57	70.3	70.2				30.185	30.478
06-26-88	0: 8:55	57..5	70.3	69.8				30.174	30.446
	12: 8:56	58	70.3	69.4				30.174	30.492
06-27-88	0: 8:56	58..5	70.7	69				30.3	30.882
	12:11:24	59	70.5	68	.368	.367	.075	30.53	30.732
06-28-88	0:11:24	59..5	70.5	67.8				30.528	30.686
	12:11:25	60	70.5	67.4	.356	.243	.078	30.568	30.85
06-29-88	0:11:25	60..5	70.7	67				30.614	30.794
	12:11:26	61	70.5	66.6	.605	.236	.07	30.628	30.842
06-30-88	0:11:27	61..5	70.5	66				30.663	30.884
	12: 6:51	62	70.3	65.8				30.634	30.74
07-01-88	0: 6:51	62..5	70.3	65.8				30.464	30.63
	12: 6:52	63	70.3	65.8				30.362	30.647
07-02-88	0: 6:53	63..5	70.5	65.8				30.288	30.53
	12: 6:53	64	70.3	65.6				30.267	30.605
07-03-88	0: 6:54	64..5	70.5	65.4				30.262	30.53
	12: 6:54	65	70.3	65				30.24	30.516
07-04-88	0: 6:55	65..5	70.5	64.6				30.236	30.494
	8: 6:55	66	70.3	64.4	.391	.204	.07	30.23	30.472

TEMPERATURE (setting:25-30 C)

STANDARD



REL.HUM.(setting:90-50% (2nd cycle))

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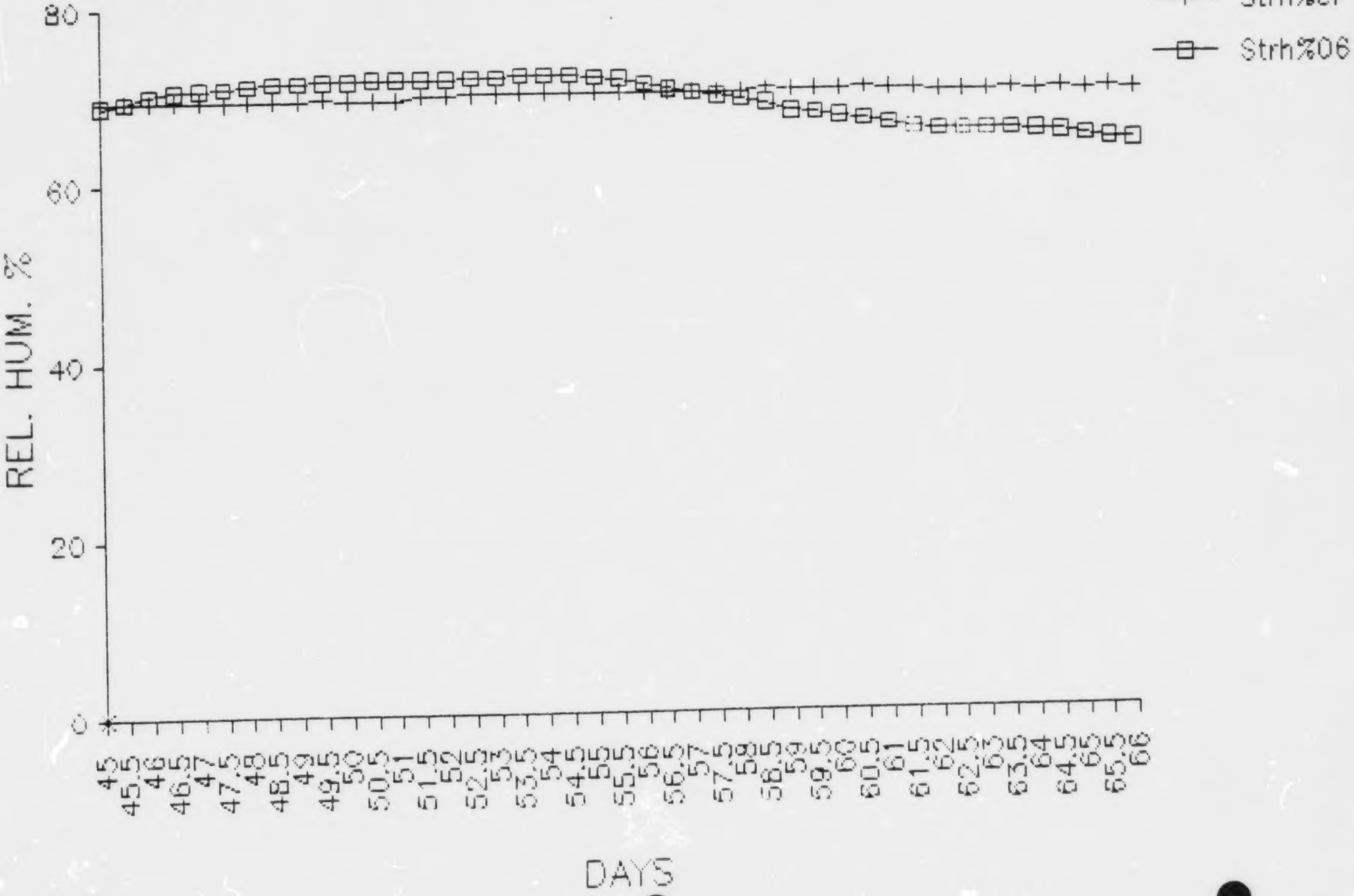

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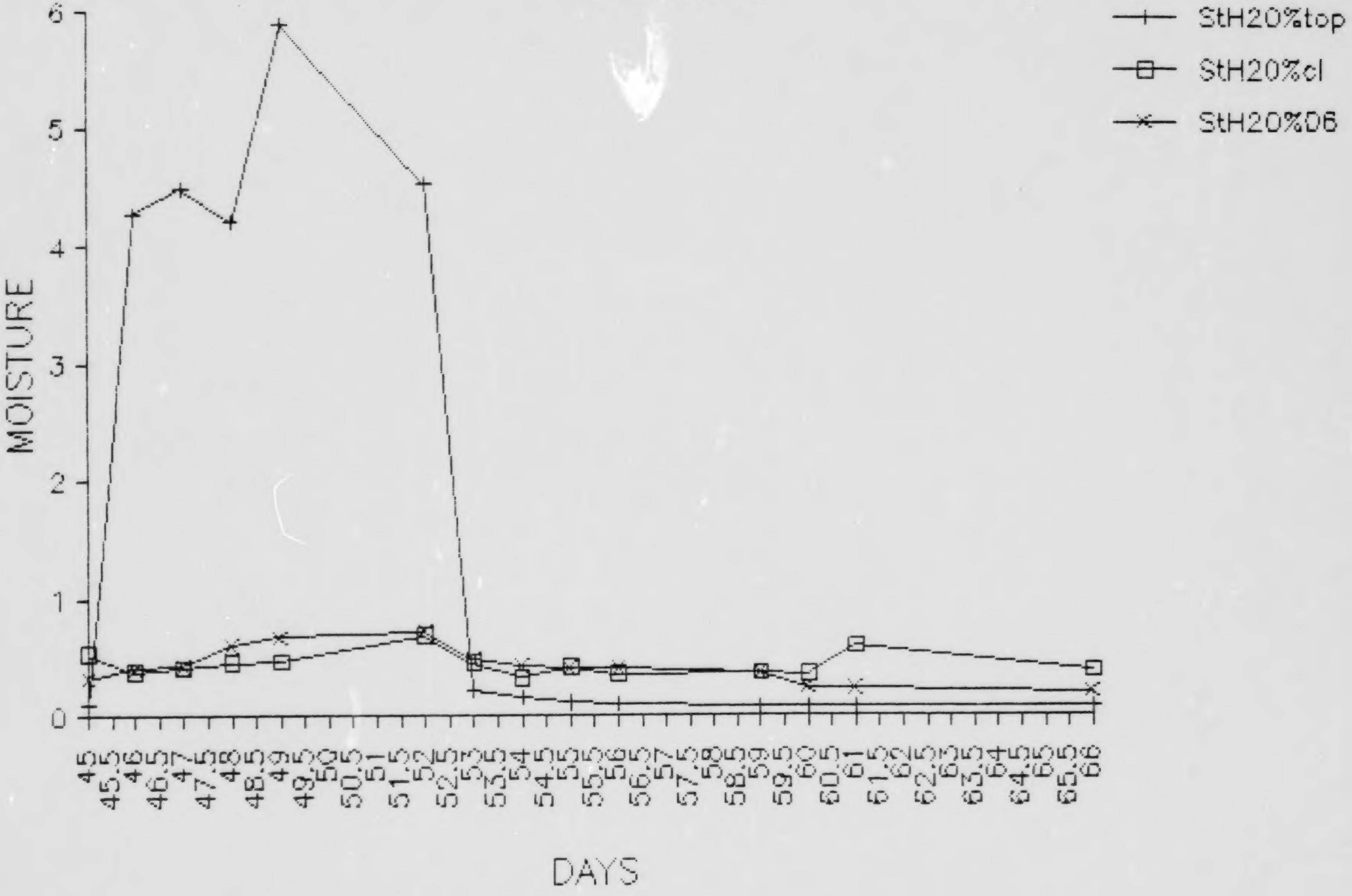

0648N

SPPA - OFFSHORE PRODUCT QUALITY: PHASE II

CHARACTERIZATION OF POTASH MATERIALS
(SUB-PROJECT 1224.03)

PROGRESS REPORT

G. G. Strathdee,
Technical Services
Potash Corporation of Saskatchewan
Saskatoon, Sask.

May 1988

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INVESTIGATION OF THE CHARACTERISTICS OF POTASH PRODUCTS FROM DEGRADATION AND CAKING TESTS

Background

High resolution Scanning Electron Microscopy (SEM) was used to compare the characteristics of potash products before and after the materials were evaluated by two standard product testing procedures. One sample of untreated, compacted SGN-210 potash, and one sample of untreated coarse natural crystal potash were run through the degradation test developed by PCS as part of the Offshore Product Quality project (1). The -8+10 mesh starting material screened from each sample, plus particles from each screen size range of the degraded potash, were observed and analyzed in the SEM so as to compare the response of each category of product in this ball mill test.

In a separate series of Anticaking Test evaluations of the effects of different reagent treatments, post-test SEM examination of SGN-210 compacted potash product was used to determine if inter-particle cementing could be detected. Samples of potash that had been exposed to a standard moisture and compression cycle typical of the product storage environment (1) were selected for analysis. Visual evidence of the post-test state of caked (or uncaked) particles was desirable to establish which physical or chemical processes contribute to set-up of potash products in storage.

Because of their prior experience in the SEM analysis of potash materials, the surface science laboratory of Atomic Energy of Canada Limited, Pinawa, Manitoba, was selected to complete this work.

Degradation Test Method and Results

A laboratory procedure and apparatus for measurement of the degradation of Granular and Coarse potash products when exposed to impact and abrasion in a ball mill have been specified for use within the potash industry (1). The procedure is included at the end of this section as Appendix 1. It is recognized that while the conditions created within the test chamber may not fully simulate those found in actual materials handling of potash products, they are reproducible. Comparisons of degradation indices calculated from the post-test particle size distribution have proven to be valuable for determination of potash product durability.

Because of the experience within the industry that compacted potash tends to be more resistant than natural crystal potash to degradation to yield fines and dust during handling, it was of interest to examine the microstructure of the screen fractions of the degraded materals to determine how and why particle breakdown occurs. Study of the two end members (100 percent natural or compacted) was considered satisfactory as degradation tests on mixtures showed that results were linearly dependent on the fraction of SGN-210 in the -8+10 mesh range.

A -8+10 mesh fraction of each of the SGN-210 compacted and coarse natural crystal products from the PCS Mining Lanigan Division were run through the degradation test and indices were obtained of 7.7 and 21.5 percent (mass fraction passing through a 20 mesh screen) respectively. Natural crystal material typically yields values which are two to three times higher than those of compacted product. Screen fractions from each test were collected and forwarded for SEM analysis.

The post-test size distributions of both degraded products are shown in Table 1. The primary fracture of natural crystal particles into the size range -10+35 mesh is clearly evident relative to the durability of the compacted product. The substantial reduction in the primary -8+10 natural crystal particles confirmed that intragranular fracture was a principal mechanism of size reduction, at least under the PCS test conditions of impact and abrasion. The view that progressive degradation of natural crystals occurs by cleavage of crystal corners, across crystal planes, was not supported by the test results. Note that below 28 mesh, the natural crystal material yielded about three times the quantity of fines and dust on any screen compared to the untreated SGN-210 product tested.

TABLE 1. Screen Analysis of Natural Crystal and Compacted SGN-210 Potash Product after Degradation Test

Screen Size	Percent Retained on Screen*			
	Natural		Compacted	
	Ind.%	Cum.%	Ind.%	Cum.%
8	0.5	0.5	7.6	7.6
10	37.3	37.8	63.5	71.1
14	27.8	65.7	16.2	87.4
20	12.8	78.5	4.9	92.3
28	7.4	85.9	2.6	94.8
35	5.1	91.0	1.8	96.6
48	3.0	94.0	1.0	97.7
65	1.8	95.8	0.6	98.3
100	1.4	97.2	0.5	98.8
-100	2.8	100.0	1.2	100.0

* Ind.%: Percent of product retained on each specified screen.
Cum.%: Cumulative percent of product retained on specified screen.

Analysis of Potash Particles in the Scanning Electron Microscope (SEM)

From potash samples delivered to the surface analysis laboratory of Atomic Energy of Canada Limited, Pinawa, Manitoba, particles were selected and mounted, vapour coated with 8 nm of gold to make the insulated KCl conductive, and examined in an ISI DS 130 Scanning Electron Microscope. A Tracor Northern EDX Detector was used for qualitative energy dispersive x-ray EDAX analysis of surface elemental composition.

The technician had previous experience in the SEM-EDAX study of potash materials and used her judgment to provide representative photographic records of the surfaces of each size range of particles. The following images are therefore assumed to be typical of the samples delivered to the AECL laboratory.

The micrographs are identified by the four digit number at the right hand extreme of the footnote code. The electron beam energy in kilovolts (kV), the magnification in power (e.g. 700X), and the dimension of the reference scale bar in microns are also shown. Some surface features that are labelled on the face of plates were analyzed using the x-ray accessory so as to qualitatively determine elemental composition. The x-ray spectra for some of these locations are included in the plates shown below.

Illustrations referred to in the following text are specified by the Figure Plate Number and the SEM micrograph registration, e.g. (1, 8066), as a convenient notation.

Comments provided by the analyst are attached as Appendix 3.

Post-Test Surface Analysis of Compacted Granular SGN 210 Product

Previous SEM studies of potash materials have been directed at understanding the internal structure of potash particles. Consequently, the present observations of the surface state of potash granules are new and any conclusions should be applied with caution. Results may not be typical of potash products processed by other methods, such as in a fluidized bed drier.

The pre-test -8+10 compacted particles had rough surfaces which probably resulted for several reasons including: impact and abrasion in the dry end of the plant; deposition of dissolved salts from process brine on to core particles during drying; product glazing; atmospheric and occluded moisture inducing surface recrystallization, perhaps influenced by the high vacuum techniques of this work; and collection of debris, fines and dust from within the process. The similar appearance of these compacted particles, and the natural crystal product discussed in the next section, suggests that the surface state is not just an artifact of these experiments.

At high magnification (see 1, 8065 and 8068) submicron sized debris can be seen. In (2, 8070) an example of a well formed microcrystal of sodium chloride, presumably from drying of process brine or a secondary recrystallization of impurity on the surface, is clearly shown. Some surface features suggest that abrasion of the surface yields scouring patterns (1, 8068). The EDAX spectra for the areas labelled J, K and L are presented on Plate 2.

After the degradation test, the residual -8+10 compacted particle surface is essentially unchanged (4, 8073-75). More submicron sized debris may be present on the unfractured surface. One cluster of well-formed, 5 to 10 micron-sized NaCl crystals was observed (area m; 3, 8071-72). These may have formed after the compaction stage as there is little apparent damage to these microcrystals. The presence of insol debris (see n; 5, 8078) containing Ca and Si suggests that transport of contaminant debris occurs easily under abrasive conditions in the degradation test.

Where fresh fracture surfaces of -8+10 particles are exposed, the transgranular cleavage of compacted particles can be seen (6, 8079 and 8080). Highly irregular KCl surfaces with little debris present indicate that ball mill impact causes random failure through compacted grains. Two types of residual porosity are evident in (6, 8079). Both larger pores from incomplete compaction, and submicron cavities from fracture through brine-filled inclusions can be seen.

All particles below 10 mesh are products of the degradation test, and in (31, 8163 to 8166) both old exterior (30, 8161 to 8162) and new fractured core surfaces can be compared. The consequence of inadequate compaction is evident in Plate 31 where in (31, 8164) conformity of some KCl grains adjacent to 2-3 micron sized pores is observable. In (31, 8165) a ring of exposed pores apparently follows the boundary of a fractured grain. The sponge-like texture of the right central area of (31, 8166) might be a result of compaction of fines and entrained air, or a secondary release of brine.

The -35+48 particles in (34, 8174, 8175 and 8183) should all have freshly exposed crystal surfaces produced during the degradation test. Micrograph (34, 8174) illustrates the rough texture caused by random cleavage through KCl grains, and the adhesion of micron-sized debris. Flaws of two types are evident. Internal pores which open to the surface and residual intragranular fracture damage can be seen in (34, 8175 and 8183).

The highly irregular surface features of -65+100 mesh compacted potash in (23, 8136 to 39) includes fractures and debris. Micron-sized NaCl crystals are present, apparently imbedded in the particle matrix. These probably originated when process brine dried on the surface of individual particles which were subsequently fixed into the core of compacted flake. Note the distribution of both porosity and fracture flaws in (23, 8137).

The internal structure of the compacted KCl grains is evident in (22, 8141). By comparison with the 4.59 μm bar, most grains in this area are 20 μm or less so represent compacted fines or dust, or fracture and compaction of larger sized feed material. The residual 1 μm pores in this incompletely compacted sample are evident in this same micrograph.

Also of interest is the distribution of submicron-sized cavities of cubic section which undoubtedly contained brine (see 22, 8142). Fracture of this zone within an inclusion-rich KCl natural crystal grain exposed this internal structure which is characteristic of another category of flaw within the KCl. Because of the presence of debris on the adjacent surface it cannot be confirmed if the cavity contents are, in some cases, recrystallized NaCl.

The finest particles of -100 mesh collected from this degradation test appear similar to those of the larger mesh size ranges with one main difference. At -100 mesh, particles probably originate from single natural crystals. The upper surfaces of the particle shown in (26, 8149) appear to be coated with a loose deposit that may be the source of much of the micron-sized debris that is distributed over the fresh cleavage planes. The presence of NaCl contaminants suggests that these were originally outer surfaces and that process brine contributed the 1-2 μm NaCl crystallites evident at points labelled "m" in (26, 8150 and 8151). In comparison, the debris in (27, 8152 and 8153) was KCl rich, probably fines generated during the fracture process in the ball mill.

Post-Test Surface Analysis of Natural Crystal Product

The natural crystal potash used in this experiment was collected after the flotation product drier at the Lanigan Division. Although this particular material is not marketed by PCS in this form, it was selected as a typical example of this type of potash which may be blended into mixtures containing compacted material of a similar size. The response of the natural crystal in the degradation test was of interest and the following notes highlight aspects of this behaviour.

At low magnification, a fracture surface of a -8+10 particle (11, 8095 and 8096) appears relatively clean and free of debris. In this example, the fracture surface follows a cleavage plane, and crack and step discontinuities that might influence the spreading of surface treatment reagents are evident. At higher magnification, the outer surface common to many particles was found to be coated with a porous deposit of evaporated process brine and insolubles (10, 8092 to 8094), similar to that observed on compacted material. Some well-formed microcrystals of 3-4 μm cross-section were present on most samples. The distinct interlocking grains in the surface layer (see 9, 8089 to 8091) suggest that the brine evaporation, or secondary crystallization affected by atmosphere moisture exchange, processes contribute to the structure of this interface. Unlike compacted potash, NaCl microcrystals would not be expected to appear on a freshly fractured natural crystal KCl surface unless transported from another location.

After the degradation test, residual -8+10 particles showed transgranular fractures characteristic of a failure under impact. Extremely rough surfaces were seen (8, 8086 to 8067). The old exterior surfaces retained the evaporated deposit (7, 8081 to 8083; 8, 8084 to 8085) although there was some evidence of localized impact or scouring (e.g. 7, 8083; 8, 8087). Loose debris was present on cleaved faces, suggesting that extremely fine particles are transported during the degradation test.

The sub 10-mesh product fractions were similar in appearance to parent -8+10 particles. In general the fines had both original crud-coated surfaces and fresh cleavage planes exposed (33, 8170 to 8173). Residual damage, in the form of incompletely propagated cracks, can also be seen in some micrographs, suggesting that further degradation of crystals would occur easily. Note the well formed 3-5 μm NaCl crystals in (32, 8169) that are well embedded in the surface deposit of evaporated process brine. This confirms that some particles in this size range were created by failure of the surface region of the initial -8+10 mesh sample.

In the smaller -35+48 mesh size ranges fewer original exterior surfaces remained (e.g. 37, 8181 and 8182) and more freshly cleaved particles were observed (36, 8178 to 8180). Debris present on the freshly exposed faces and in residual cracks (36, 8180) was probably transported to those locations after spalling and blending occurred in the ball mill test. The NaCl microcrystal detected in (36, 8179) is diagnostic of this.

Below 65 mesh the fines are generally cubical in shape (24, 8143), suggesting that parent particle failure involves crystal break-up and not just shear at sharp corners as has been proposed as one mechanism of natural crystal degradation. The high resolution micrographs of the L-shaped crystal in (24, 8143) show what is probably an area of original external surface. Debris, including NaCl microcrystals, that is present on freshly cleaved KCl was undoubtedly collected during blending in the test apparatus (see 25, 8147 and 8148).

The examples of -100 mesh fines seem to include two types of particle (28, 8154). Where all exposed faces are fresh cleavage surfaces, the resulting particles certainly originated by degradation of KCl which was part of the core of the -8+10 starting material (28, 8155 and 8157). Other -100 mesh fines appear to have resulted by spallation or abrasion of the surface deposit (29, 8156, 8158 and 8159). The presence of embedded NaCl microcrystals in the latter particles distinguishes the two types of fines. Note the residual flaws in the Plate 29 particles which should facilitate further breakdown during handling to even finer air-suspendable potash.

Post-Anticaking Test SEM Analysis of Treated Potash Products

The performance of samples of a SGN-210 compacted potash product treated with different anticaking and/or dedusting reagents were evaluated in the standard anticaking test described in Appendix 2. Potash plugs recovered by destructive extrusion from the pressed pellets were examined in the SEM in an attempt to observe interparticle recrystallization or other changes at contact points that may have contributed to product set-up when moisture, temperature and compression load were varied during the test.

Table 2 lists the loads applied to the destructive test piston to cause breakdown of the caked samples. The untreated SGN-210 product set up hard, however the three treated samples behaved similarly, all requiring breaking loads in the range 370 ± 20 pounds. Granular particles in the range -6+10 mesh were used in the anticaking test. Some of the microstructure evident in the following SEM micrographs originated with secondary processes.

TABLE 2. Destructive Testing of Caked Potash Pellets*

Reagent Treatment	Piston Load at Failure
Untreated	850 ± 100
Arosurf - Canola	385
Armac T - Husky	350
Bardust	357

* See Appendix 2 for the anticaking test method.

The untreated product surface at low magnification was similar in appearance to those observed in degradation tests. Surface roughness was characteristic of a process brine deposit. However, the enhanced etching of individual grain boundaries and the more rounded features (see 20, 8128 to 8130), combined with the occurrence of well-formed sharp-edged NaCl microcrystals suggests that secondary dissolution-recrystallization was an important surface process. In addition there was evidence of trans-granular fracture of KCl microcrystals (21, 8135) which had formed at interparticle contacts during the caking stage of the test. The cluster of rounded KCl crystals shown in (21, 8133) also supports the case for a recrystallization cementing mechanism as being the principal reason for product set-up. This appears to be dominated by formation of a network of KCl crystals of ≅ 20 μm size, with associated non-structural NaCl.

The surface of potash treated with Armac-T and Husky S288 dedusting oil exposed to the temperature and humidity cycle of the test was porous and consisted of a deposit of highly irregular KCl (k) and micron-sized NaCl (m) (18, 8124 and 19, 8125). The size distribution of NaCl microcrystals was 1-15 μm (19, 8125), perhaps greater than that previously observed in degradation test material. This suggests that secondary recrystallization is a generally distributed phenomenon during caking, and not limited to interparticle contacts. The clean fracture surfaces (19, 8126 and 8127) are free of the usual debris and indicate that failure has occurred through an original particle matrix. The interparticle cementing presumably was stronger than the KCl area under observation. Note the residual flaws or porosity present at this exposed surface, and the absence of the usual NaCl microcrystals.

The post-test analysis of product treated with Arosurf MG 102 W plus Canola oil is shown in micrographs 8114 to 8121. Extensive surface recrystallization of both KCl and NaCl is evident in all areas. The localized insol (r in 15, 8115) did not seem to play any role in caking, although it may have been exposed by local dissolution/crystallization. It is possible that the smooth patches seen in (16, 8117 to 8119) are islands of reagent deposited during product treatment. These would not yield an x-ray fluorescence spectrum because of the low atomic number of the constituent elements. The fracture surfaces in (17, 8120 and 8121) are like those trans-particle equivalents seen in degraded product.

The surface of Bardust (a waxy additive) treated material generally appeared more amorphous in microstructure than that from other reagent treatments. Secondary recrystallization was extensive. No clear cut evidence of cementing bridges was provided, however the principal trans-particle fracture pattern (14, 8112 and 8113) suggests that interparticle bonds were strong. The striated patches in (13, 8109) may be islands of Bardust as they do not contain the usual surface debris. Note the extremely small NaCl microcrystals adjacent to the edge of such a patch in (13, 8110), and the isolated single crystal of NaCl in (12, 8107).

Conclusions from SEM Analysis

- Examination of the screened fractions of potash after degradation tests on both natural crystal and compacted potash products confirms that natural Coarse material is more sensitive to impact and abrasion because of the relative ease of intracrystalline fracture. In comparison, the random distribution of KCl grains in compacted material, and the pinning effect of microcrystalline NaCl dried on to compactor feed, yield a more durable matrix which is more resistant to degradation. Residual flaws in compacted product which are present as brine inclusions in grains, or porosity at grain boundaries, probably determine the mechanism of failure of compacted particles.

- The porosity and roughness of the weathered surfaces of both natural crystal and compacted potash product are comparable. This condition probably originates with the evaporation of the dissolved salts in process brine on to exposed surfaces in the drying stage. Freshly broken potash surfaces exhibit well-defined fracture planes characteristic of single crystals of KCl. Some debris from the overall degradation process is also redistributed on to freshly fractured faces but this can be distinguished from an interface that has had a moisture cycle.

  The consequence of this surface roughness is that liquid phase reagents, even those with low contact angles on potash (1), will not spread readily over potash with a recrystallized surface. Additional investigation will be required to determine the effect of processing equipment and practices such as glazing, on the morphology of this surface deposit. The presence of microcrystalline NaCl is one indication that the layer is formed upon evaporation of process brine containing KCl and NaCl solutes. Variations in the characteristics of this layer may account for the different reagent loadings required to achieve a specified level of dedusting or anticaking performance.

  The surface deposit may also contribute to dustiness of potash materials by release of air-suspendable micron-sized particles upon abrasion, without the need for progressive degradation of potash particles down to that size range. The importance of this mechanism should be evaluated by retesting potash product prepared by the addition of tracer cations (Rb+, CS+) to process brines before product drying.

- Although definitive SEM evidence was not collected on samples of intentionally caked potash material, the micrographic results were consistent with interparticle cementing being dependent on surface dissolution and recrystallization at contacts. The SEM technique will not detect organic reagents directly because of the low atomic number of the elements C, H, N. However, vapour deposition of gold on to the organic films should reveal images of any liquid phase present on coated particles. There was some evidence that incomplete reagent spreading had occurred, as a consequence of the intrinsic surface roughness of the potash used in these experiments.

APPENDIX 1

DEGRADATION TEST PROCEDURES

CANPOTEX BALL MILL DEGRADATION TEST STANDARD FOR GRANULAR OR COARSE PRODUCT

EQUIPMENT

1. A stainless steel ball mill container, 7.5 inches in diameter by 2.5 inches wide, with 6 flights 0.5 inches high, equally spaced around the internal circumference of the chamber.

   The ball mill is driven by a gear motor at 33.4 rpm.

2. 25 steel balls, 0.5 inches in diameter, total weight of 208.2 grams. The balls should be stored in methanol when not in use to prevent rusting.

3. Tyler mesh screens full height stainless steel 8 mesh, 10 mesh, 20 mesh and a scalping screen larger than 8 mesh but smaller than 0.5 inch. These screens should be reserved for degradation tests and should be washed periodically to prevent build-up of reagents and potash.

4. Rotap* shaker with a hammer.

5. Automatic timer to start and stop equipment in a 5.0 minute test interval.

PROCEDURE

1. Split the representative potash sample to obtain 100 to 150 grams of -8+10 mesh material. Screen on the Rotap with the hammer down using the 8 and 10 mesh screens for 5.0 minutes.

2. Weigh 100 grams of the -8+10 mesh productand transfer it to the ball mill container. It is essential that exactly 100 grams of product ends up in the ball mill container.

3. Add the 25 steel balls.

4. Rotate the ball mill for 5.0 minutes at 33.4 rpm.

5. Transfer the contents of the ball mill container to the scalping screen. The scalping screen is placed on top of the 20 mesh screen to retrieve the steel grinding balls.

6. Remove the scalping screen. Screen the contents of the 20 mesh screen on the Rotap shaker for 2.0 minutes with the apparatus hammer down.

7. Weight the contents of the 20 mesh screen.

8. Calculate (Percent Degradation) = (100%) - (Mass in grams of potash product retained on 20 mesh screen) = Degradation Index.

---

* Rotap is the trade name of a screen shaker apparatus Testing Sieve Shaker Model B supplied by the C.E.Tyler Company.

APPENDIX 2

ANTICAKING TEST PROCEDURE

ANTICAKING PROCEDURE STANDARD PRODUCT

PROCEDURE

1. Reagentize the product in a heated mixer keeping the temperature of the product between 60°C and 70°C. Mix for 15 minutes.

2. Store the reagentized product in glass jars for 4 weeks.

3. Moisturize 70 grams of product for 6 hours in the desiccator using the anticaking jars (Appendix 7). The dessicant is a saturated solution of sodium hydrogen orthophosphate ($Na_2HPO_4.12H_2O$).

4. Load the samples into the anticaking presses (Appendix 2) with 60 grams of the moisturized product and leave in the presses for 1 day at 40 psi and room temperature. Do a gravimetric moisture test at 70°C on the remaining 10 grams of product.

5. After 1 day in the presses, turn the heat on in the anticaking cabinet (40°-45°C) and leave on for 1 day.

6. Remove the molds from the presses and break the resulting briquettes on the carver press using the die which punches a hole through the briquette, and record lbs required to break.

DEDUSTING PROCEDURE - STANDARD PRODUCT

1. Transfer 200 grams of product to the dedusting pot along with fifty (50) 5/16" steel balls.

2. Weigh the filter apparatus.

3. Attach the filter to the vacuum pump and adjust the vacuum to 15 psi.

4. Rotate for 15 minutes with vacuum pump running.

5. Weigh the filter apparatus and calculate ppm dust.

6. Wash the pot with methanol after tests with reagentized products.

APPENDIX 3

AECL SEM ANALYSIS REPORT

APPENDIX 3

ATOMIC ENERGY OF CANADA LIMITED
Whiteshell Nuclear Research Establishment

ANALYTICAL SCIENCE BRANCH

AS-54092

ANALYSIS REPORT

Sample Description: Potash samples (Kalium Chemicals)

	Code	Time
Instrument:	ISEM	11 h
Person:	DR	7 h
	G_1	26.5 h
	G_1 (Alan)	5 h
Branch:	COMM.	
W.P. Code:	9365-0	

Submitted by: Joe Gosselin   Badge #:   No. of Analysis: 111

RESULTS: Compacted Granular -8 + 10 not degraded (54092-1)

Micrographs 8065 to 8067 of Figure Plate 1 are increasing magnification micrographs of an area typical of the C.G. -8 + 10 crystals. Several sulphur containing particles (J) were noted on the surface (see spectrum 'K', Figure Plate 2). The majority of the particles seen here were rich in potassium and chlorine as was the background (k), see spectrum 'k'). Micrograph 8068 is an overview of another crystal with a similar surface structure, however, no sulphur containing particles were found.

Micrographs 8069 and 8070 of Figure Plate 2 are of a portion of a fractured C.G. -8 + 10 crystal. Several sodium and chlorine rich crystals (l) were found on this fractured surface (see spectrum 'l').

Compacted Granular -8 + 10 degraded (54092-2).

These crystals were found to have patches of sodium and chlorine rich crystals (m), (see spectrum 'm'). Micrographs 8071 to 8075 of Figure Plates 3 and 4 showing several of these patches on different crystals. The surrounding areas and smaller particles were all found to be rich in potassium and chlorine as expected (see spectrum 'k').

Micrographs 8076 to 8078 of Figure Plate 5 are of another C.G. -8 + 10 degraded crystal which did not show evidence of the NaCl clustering as seen on the other crystals. Only a few NaCl crystals (m) were noted. This crystal was also found to have several calcium rich particles (n) resting on its surface as seen in micrograph 8078 (see spectrum 'n').

Micrographs 8079 and 8080 of Figure Plate 6 are of a portion of the fractured surface of one of the C.G. -8 + 10 degraded crystals typical of the rest. There was no evidence of NaCl crystals or any other crystals other than KCl.

Lanigan Natural Coarse -8 + 10 degraded (54092-3)

Micrographs 8081 to 8083 of Figure Plate 7 are increasing magnification micrographs of an area typical of the surface of the L.N.C. -8 + 10 degraded crystals. Some NaCl crystals (m) were noted across the surface. Only one crystal of those analyzed was found to have NaCl clusters (O) similar to those seen of C.G. -8 + 10 degraded. Micrographs 8084 and 8085 of Figure Plate 8 are of this cluster (O).

Micrographs 8086 and 8087 are of a fracture surface typical of the L.N.C. -8 + 10 degraded crystals. There were no NaCl crystals or other unusual precipitates found.

Lanigan Natural Coarse -8 + 10 not degraded (54092-4)

Micrographs 8089 to 8090 of Figure Plate 9 are increasing magnification micrographs of an area typical of the surface of one of the L.N.C. -8 + 10 crystals. Many NaCl crystals (m) and sulphur containing particles (p), (see spectrum 'p') were found here. The majority of the L.N.C. -8 + 10 crystals did not have as rough a surface as this crystal. Micrographs 8092 to 8094 of Figure Plate 10 are of an area typical of the surface of the majority of the crystals. NaCl crystals (m) and sulphur containing particles (p) were found on these surfaces as well.

Micrographs 8095 and 8096 of Figure Plate 11 are of a typical L.N.C. -8 + 10 crystal fracture surface. No unusual crystal forms were found.

Bar Dust (54092-21)

Micrographs 8106 to 8111 of Figure Plates 12 and 13 are varying magnification micrographs of typical Bar Dust crystals. A small precipitate (q) containing iron (see spectrum 'q') was found on one of the crystals (see micrograph 8108). Many NaCl crystals (m) were seen across the surface. It appeared that there was a film of small forming crystals (see micrographs 8108, 8110, and 8111). Some were found to be NaCl (m) and others were KCl like the background larger crystals (k).

Micrographs 8079 and 8080 of Figure Plate 6 are of a portion of the fractured surface of one of the C.G. -8 + 10 degraded crystals typical of the rest. There was no evidence of NaCl crystals or any other crystals other than KCl.

Lanigan Natural Coarse -8 + 10 degraded (54092-3)

Micrographs 8081 to 8083 of Figure Plate 7 are increasing magnification micrographs of an area typical of the surface of the L.N.C. -8 + 10 degraded crystals. Some NaCl crystals (m) were noted across the surface. Only one crystal of those analyzed was found to have NaCl clusters (O) similar to those seen of C.G. -8 + 10 degraded. Micrographs 8084 and 8085 of Figure Plate 8 are of this cluster (O).

Micrographs 8086 and 8087 are of a fracture surface typical of the L.N.C. -8 + 10 degraded crystals. There were no NaCl crystals or other unusual precipitates found.

Lanigan Natural Coarse -8 + 10 not degraded (54092-4)

Micrographs 8089 to 8090 of Figure Plate 9 are increasing magnification micrographs of an area typical of the surface of one of the L.N.C. -8 + 10 crystals. Many NaCl crystals (m) and sulphur containing particles (p), (see spectrum 'p') were found here. The majority of the L.N.C. -8 + 10 crystals did not have as rough a surface as this crystal. Micrographs 8092 to 8094 of Figure Plate 10 are of an area typical of the surface of the majority of the crystals. NaCl crystals (m) and sulphur containing particles (p) were found on these surfaces as well.

Micrographs 8095 and 8096 of Figure Plate 11 are of a typical L.N.C. -8 + 10 crystal fracture surface. No unusual crystal forms were found.

Bar Dust (54092-21)

Micrographs 8106 to 8111 of Figure Plates 12 and 13 are varying magnification micrographs of typical Bar Dust crystals. A small precipitate (q) containing iron (see spectrum 'q') was found on one of the crystals (see micrograph 8108). Many NaCl crystals (m) were seen across the surface. It appeared that there was a film of small forming crystals (see micrographs 8108, 8110, and 8111). Some were found to be NaCl (m) and others were KCl like the background larger crystals (k).

Micrographs 8104 and 8105 of Figure Plate 14 are of another Bar Dust crystal. Very few NaCl crystals were found on its surface.

Micrographs 8112 and 8113 are of a fracture surface typical of the Bar Dust crystals. No unusual crystals forms were seen.

Arosurf MG 102 W

Canola - ~~Arosart Mg 102~~ W (54092-22)

Micrographs 8114 to 8119 of Figure Plates 15 and 16 are varying magnification micrographs of the surfaces of typical Canola crystals. A patch containing a thin layer of silicon, aluminum, and magnesium precipitate (r), (see spectrum 'r') was noted on one of the crystals (see micrographs 8114 and 8115). Clusters of NaCl crystals (m) were noted on all the crystals examined.

Micrographs 8120 and 8121 of Figure Plate 17 are of a fracture surface typical of these crystals. No unusual crystal forms such as NaCl were found.

Husky - Armac T (54092-23)

Micrographs 8122 to 8125 of Figure Plates 18 and 19 are varying magnification micrographs of surfaces typical of the Husky-Armac T crystals. These crystal surfaces were found to be littered with large and small NaCl crystals (m). The NaCl crystals appeared to be located in the larger KCl crystal crevices or at the KCl crystal boundaries. This effect was also noted on the Bar Dust and Canola-Arosart Mg 102W crystals but to a lesser extent.

Micrographs 8126 and 8127 of Figure Plate 19 are of a fracture surface typical of the Husky Armac-T crystals. No unusual crystals forms were found.

No Treatment (54092-24)

Micrographs 8128 to 8132 of Figure Plates 20 and 21 are varying magnification micrographs of surfaces typical of the No Treatment crystals. Many large and small NaCl crystals (m) were found protruding from the surface. Micrograph 8129 shows a precipitate film (s) found on a portion of one of the crystals. This film was found to contain silicon, sulphur, and calcium (see spectrum 's').

Micrographs 8133 and 8135 of Figure Plate 21 are of a "fractured" surface of one of the crystals. This sample of crystals did not fracture in the same way as the others did. It appeared to "crumble" apart rather than fracture. NaCl crystals (m) were found on the exposed KCl crystal surfaces.

Compacted Granular -65 + 100 (54092-17)

Micrograph 8140 of Figure Plate 22 shows three crystals typical of the types found in sample C.G. -65 + 100. Micrograph 8141 is a closer view of the larger crystal. NaCl crystals (m) were found at the KCl crystal boundaries. Micrograph 8142 is a close view of the flatter crystal. There were no NaCl crystals found here. All the particles present were rich in potassium and chlorine. Micrographs 8136 to 8139 of Figure Plate 23 are of two other crystal surfaces. A few NaCl crystals (m) were seen on their surfaces.

Lanigan Natural Coarse -65 + 100 (54092-18)

Micrograph 8143 of Figure Plate 24 shows a group of crystals typical of the types found in sample L.N.C. -65 + 100. Micrograph 8144 is a close view of the flatter type crystal surface. Micrographs 8145 and 8146 are close views of the L-shaped crystal. A few NaCl crystals were found on its surface. Micrographs 8147 and 8148 of Figure Plate 25 are close views of one of the other crystals. All of the particles present were found to be rich in potassium and chlorine.

Compacted Granular -100 (54092-19)

Micrographs 8149 to 8153 of Figure Plates 26 and 27 are varying magnification micrographs of crystal surfaces typical of the C.G. -100 sample. A few NaCl crystal were found on some of the crystal surfaces (see micrograph 8150). The majority of the crystals of this sample appeared similar to the one seen in micrograph 8152. The particles resting on these surfaces were found to be rich in potassium and chlorine.

Lanigan Natural Coarse -100 (54092-20)

Micrograph 8154 of Figure Plate 28 shows a group of crystals typical of the types found in sample L.N.C. -100. Migrographs 8155 to 8159 of Figure Plates 28 and 29 are close views of these crystals. A few NaCl crystals were noted on the surface of the crystal seen in micrographs 8158 and 8159. All the loose particle debris was found to be rich in potassium and chlorine.

Compacted Granular -10 +14 (54092-5)

Micrographs 8160 to 8164 of Figure Plates 30 and 31 are varying magnification micrographs of surfaces typical of the C.G. -10 + 14 crystals. Some small NaCl crystals were noted across the surfaces. Micrographs 8163 and 8164 show an area where many NaCl crystals were found on "exposed" KCl crystals. Several other areas similar to this were found.

Compacted Granular -65 + 100 (54092-17)

Micrograph 8140 of Figure Plate 22 shows three crystals typical of the types found in sample C.G. -65 + 100. Micrograph 8141 is a closer view of the larger crystal. NaCl crystals (m) were found at the KCl crystal boundaries. Micrograph 8142 is a close view of the flatter crystal. There were no NaCl crystals found here. All the particles present were rich in potassium and chlorine. Micrographs 8136 to 8139 of Figure Plate 23 are of two other crystal surfaces. A few NaCl crystals (m) were seen on their surfaces.

Lanigan Natural Coarse -65 + 100 (54092-18)

Micrograph 8143 of Figure Plate 24 shows a group of crystals typical of the types found in sample L.N.C. -65 + 100. Micrograph 8144 is a close view of the flatter type crystal surface. Micrographs 8145 and 8146 are close views of the L-shaped crystal. A few NaCl crystals were found on its surface. Micrographs 8147 and 8148 of Figure Plate 25 are close views of one of the other crystals. All of the particles present were found to be rich in potassium and chlorine.

Compacted Granular -100 (54092-19)

Micrographs 8149 to 8153 of Figure Plates 26 and 27 are varying magnification micrographs of crystal surfaces typical of the C.G. -100 sample. A few NaCl crystal were found on some of the crystal surfaces (see micrograph 8150). The majority of the crystals of this sample appeared similar to the one seen in micrograph 8152. The particles resting on these surfaces were found to be rich in potassium and chlorine.

Lanigan Natural Coarse -100 (54092-20)

Micrograph 8154 of Figure Plate 28 shows a group of crystals typical of the types found in sample L.N.C. -100. Migrographs 8155 to 8159 of Figure Plates 28 and 29 are close views of these crystals. A few NaCl crystals were noted on the surface of the crystal seen in micrographs 8158 and 8159. All the loose particle debris was found to be rich in potassium and chlorine.

Compacted Granular -10 +14 (54092-5)

Micrographs 8160 to 8164 of Figure Plates 30 and 31 are varying magnification micrographs of surfaces typical of the C.G. -10 + 14 crystals. Some small NaCl crystals were noted across the surfaces. Micrographs 8163 and 8164 show an area where many NaCl crystals were found on "exposed" KCl crystals. Several other areas similar to this were found.

Micrographs 8165 and 8166 of Figure Plate 31 are of a fracture surface typical of the C.G. -10 + 14 crystals. No unusual crystals forms were noted.

Lanigan Natural Coarse -10 + 14 (54092-6)

Micrographs 8167 to 8169 of Figure Plate 32 are increasing magnification micrographs of one of the L.N.C. -10 + 14 crystals. This crystal was found to have many NaCl crystals on its surface. It was also one of the white crystals in the sample.

Micrographs 8170 and 8171 of Figure Plate 33 are of a crystal surface more typical of the L.N.C. -10 + 14 crystals. There were no NaCl crystals found. The particles seen here were all rich in potassium and chlorine.

Micrographs 8172 and 8173 are of a fracture surface typical of the L.N.C. -10 + 14 crystals. No unusual crystal forms were found.

Compacted Granular -35 + 48 (54092-3)

Micrographs 8174 and 8175 of Figure Plate 34 are of a crystal typical of the type found in sample C.G. -35 + 48. Micrograph 8183 is of another crystal found to have NaCl cubes resting on its surface. Micrographs 8176 and 8177 of Figure Plate 35 are of another crystal. NaCl crystals were found on the "exposed" KCl crystals.

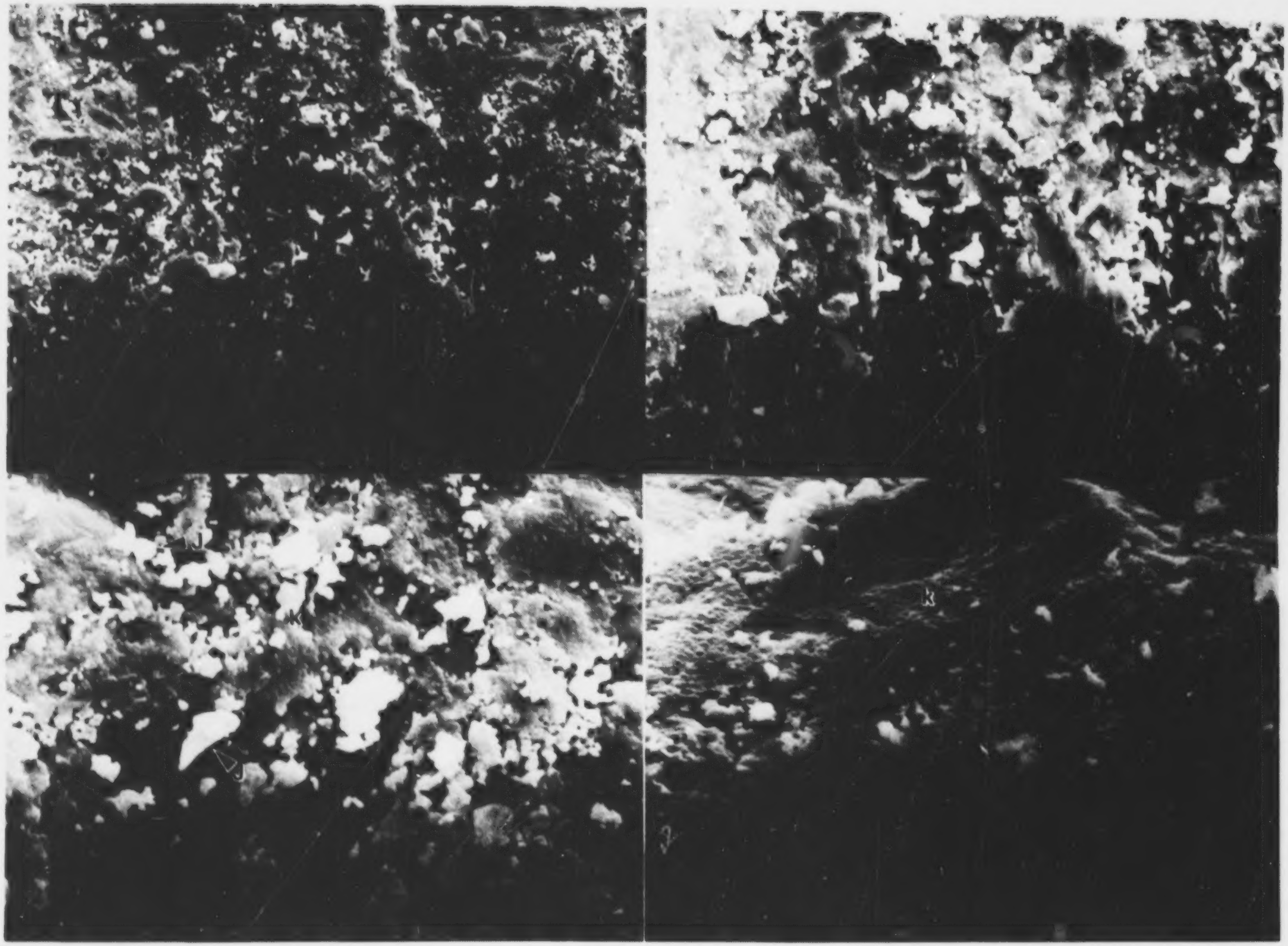
Lanigan Natural Coarse -35 + 48 (54092-14)

Micrograph 8178 of Figure Plate 36 is of a group of crystals typical of the types found in sample L.N.C. -35 + 48. Micrographs 8179 to 8182 of Figure Plates 36 and 37 are close views of the various types found. A few NaCl crystals (m) were noted on all surfaces. Clusters of NaCl crystals were found on the rougher surfaces like those seen in micrographs 8181 and 8182.

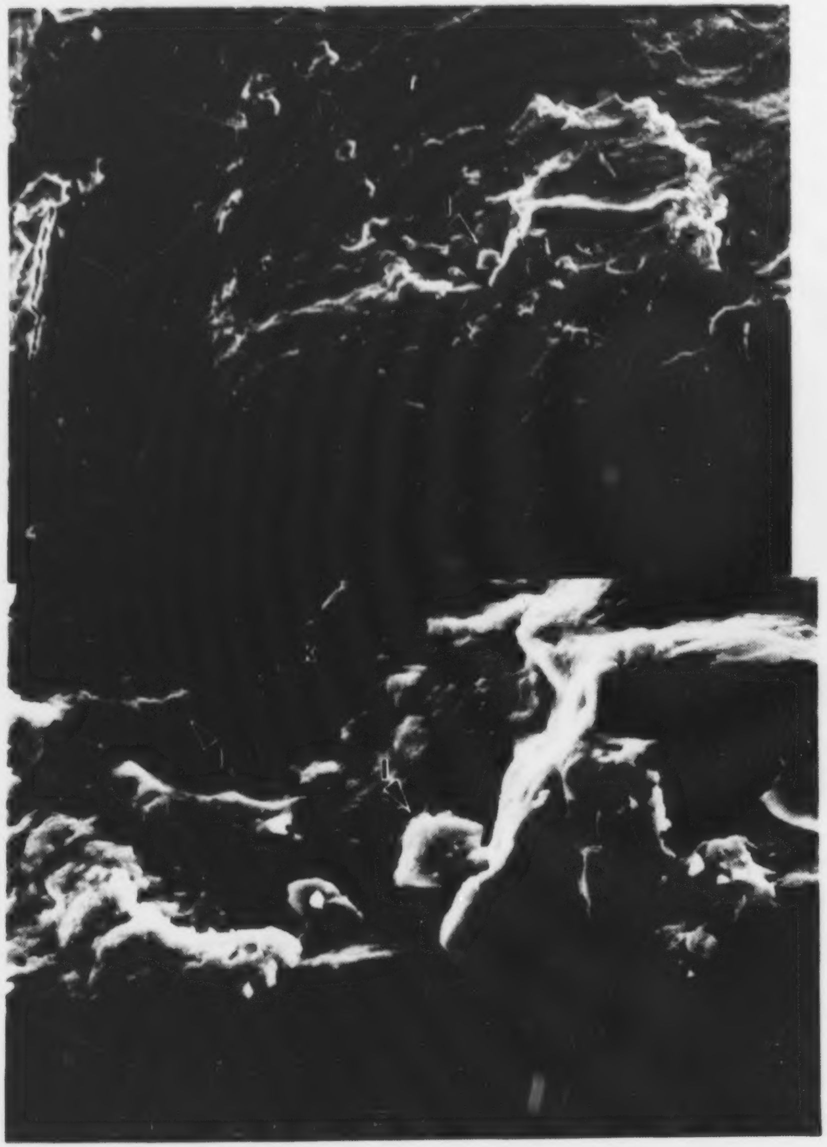
REMARKS: All gold peaks in x-ray spectra are due to the gold coating sputtered onto the sample surfaces prior to analysis in order to prevent sample charging.

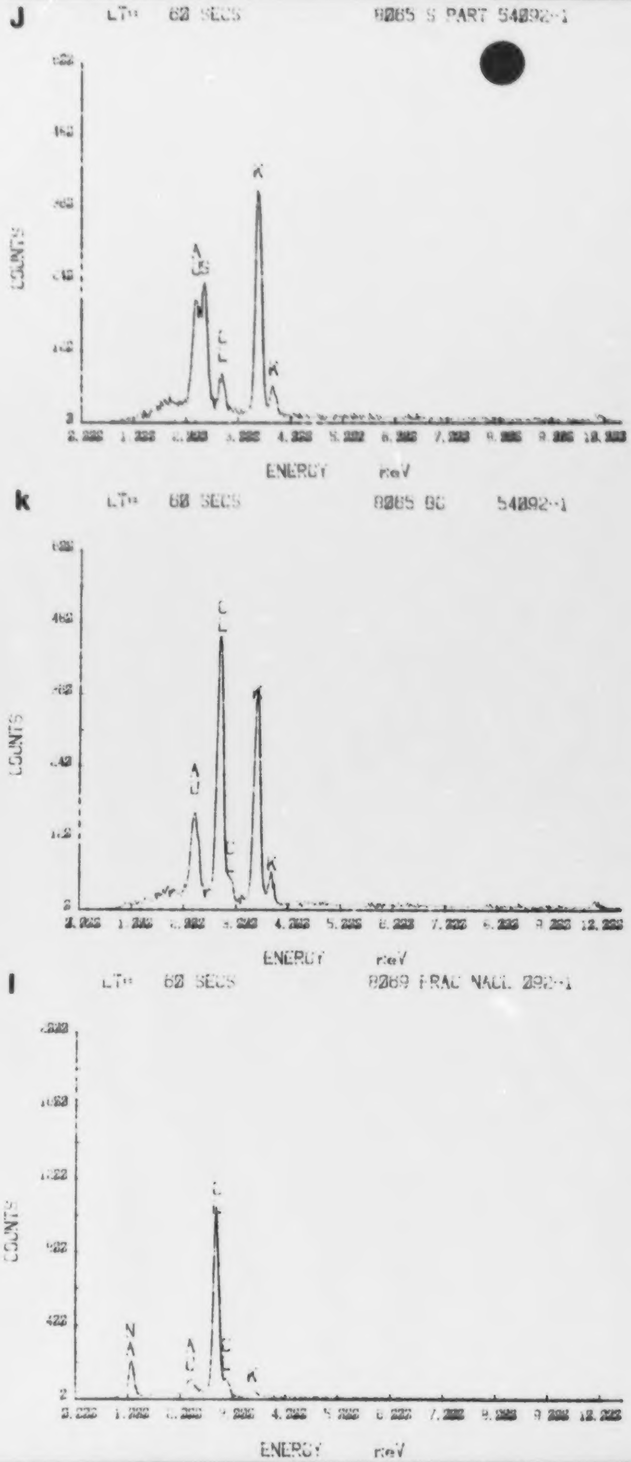
Date: 1988 March 28 Analyst(s): K.J. George

PLATES

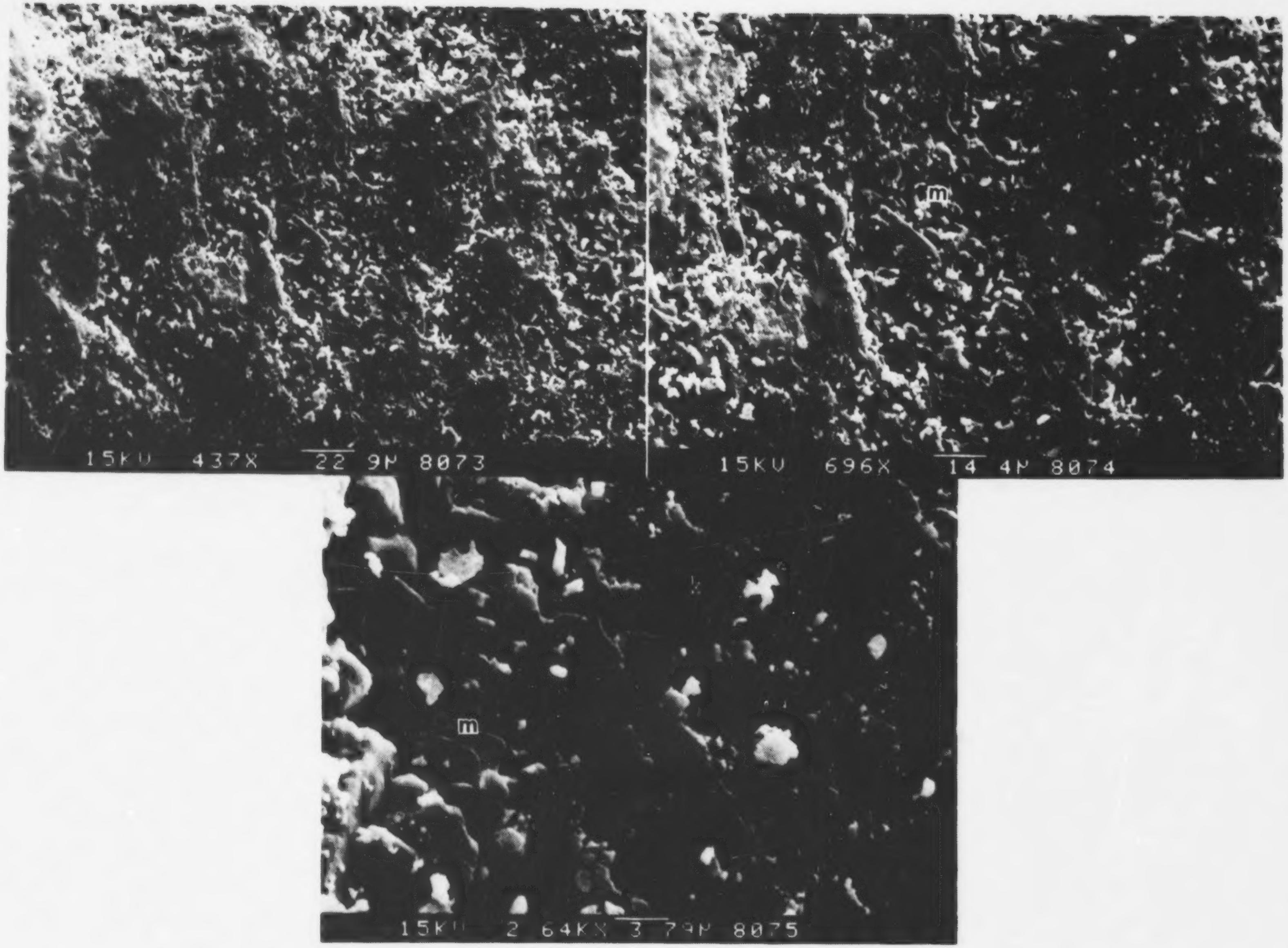


AS 54092
Figure Plate 1
Compacted Granular -8+10 Not Degrad

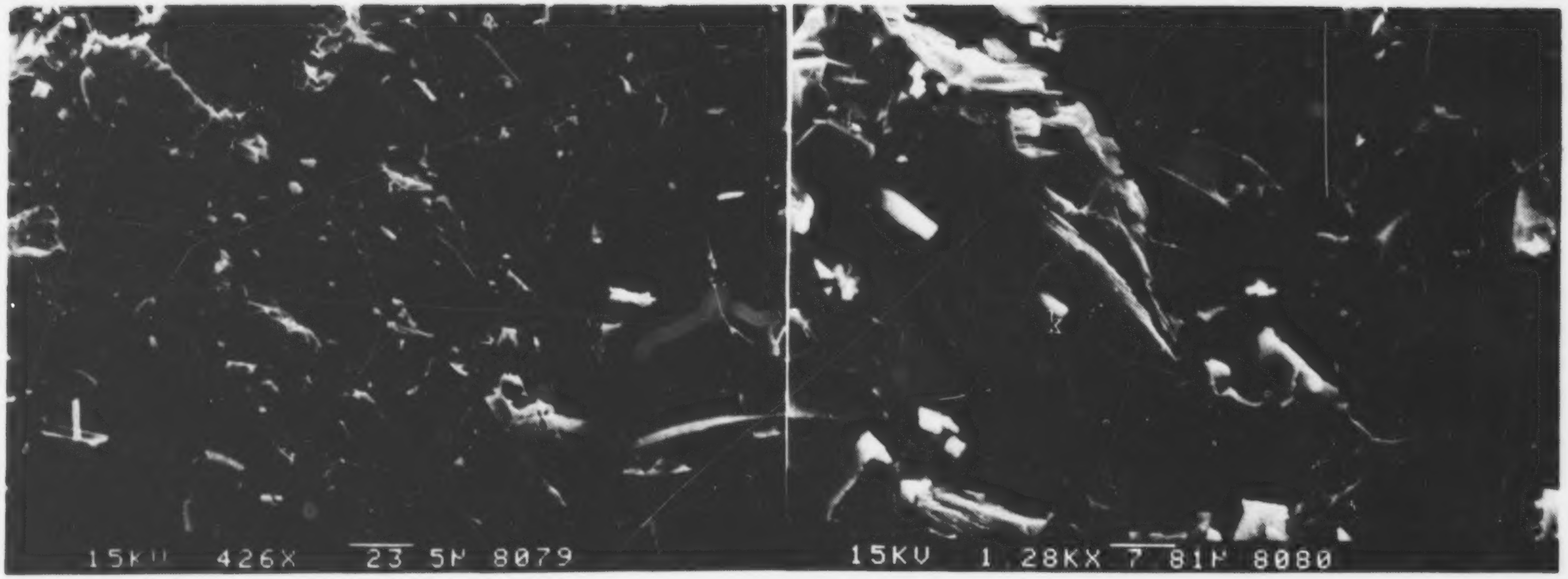




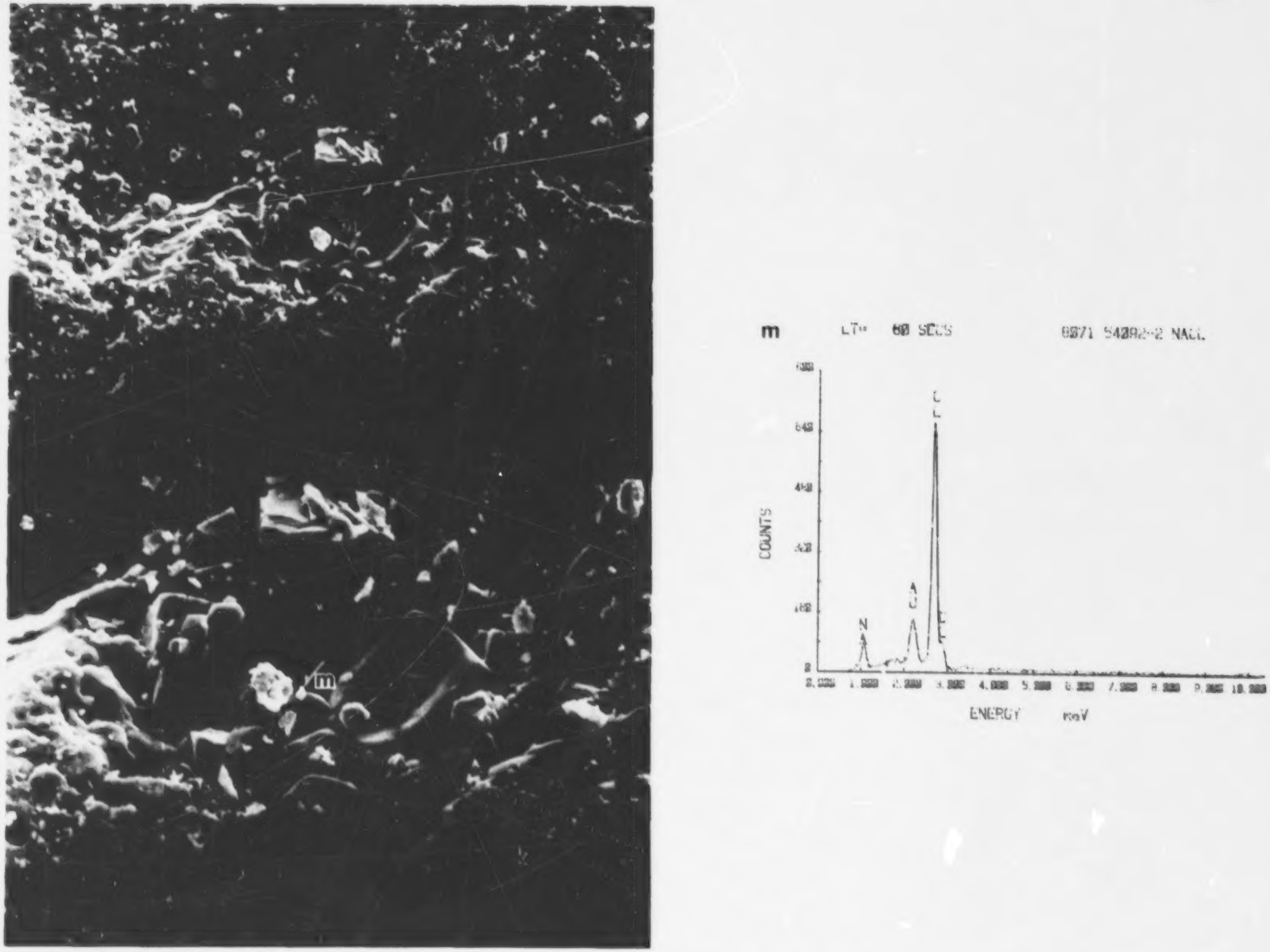
AS 54092
Figure Plate 2
Compacted Granular -8+10 Not Degrad


15KV 437X 22 9µ 8073
15KV 696X 14 4µ 8074
m
15KV 2 64KX 3 79µ 8075
m

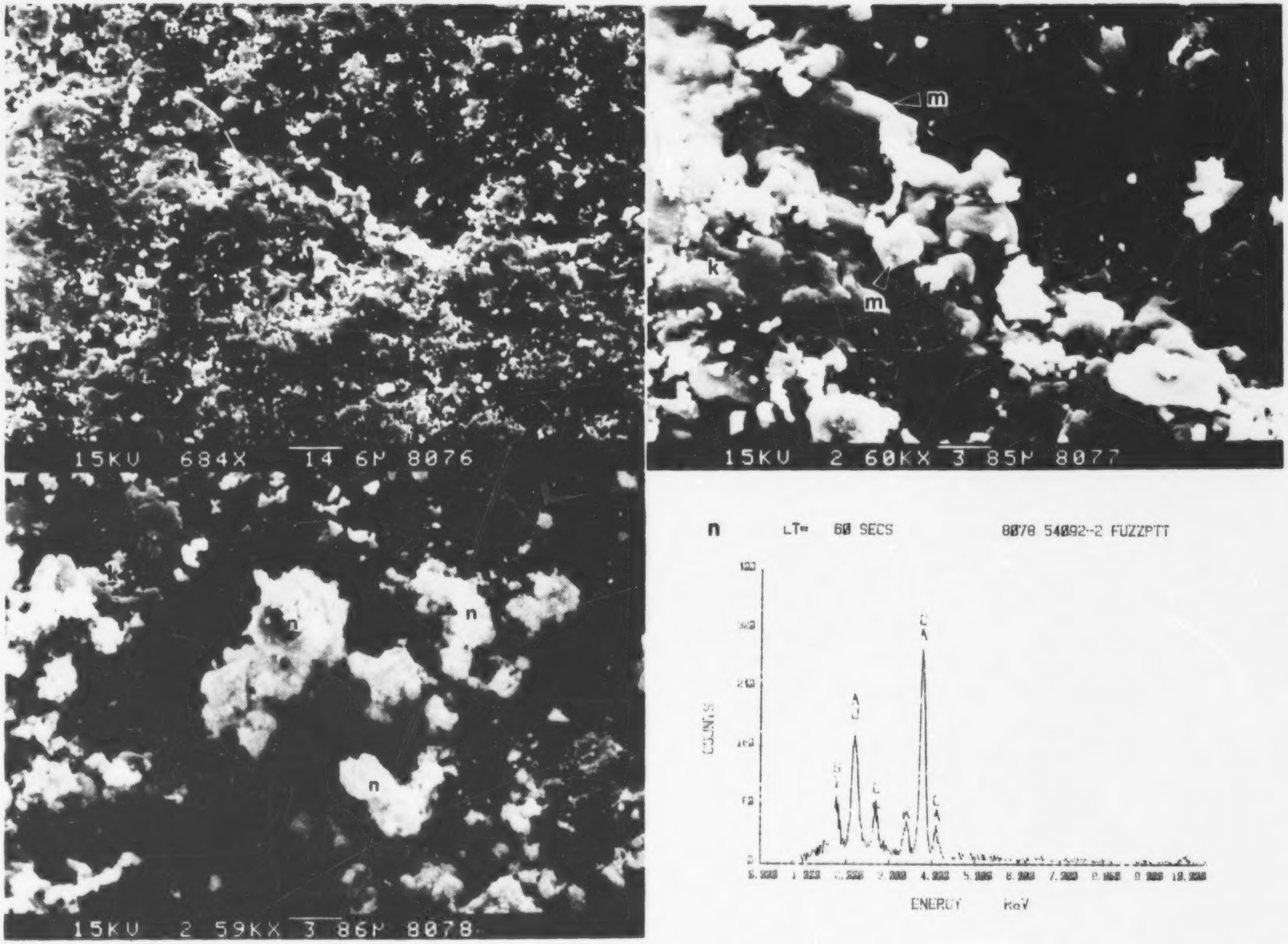
AS 54092
Figure Plate 4
Compacted Granular -8+10 Degrad


15KV 426X 23 5µ 8079
15KV 1 28KX 7 81µ 8080

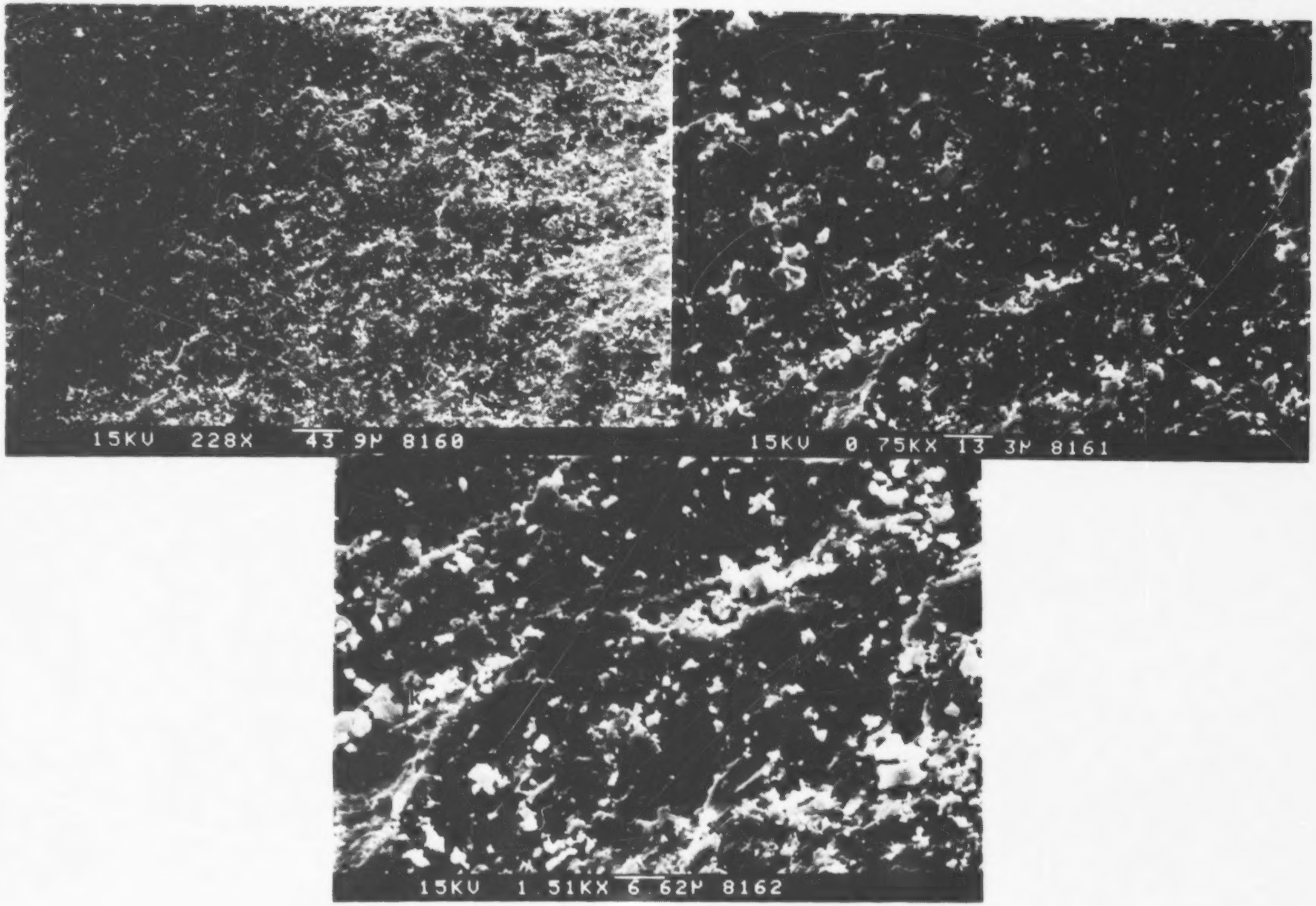
AS 54092
Figure Plate 6
Compacted Granular -8+10 Degrad



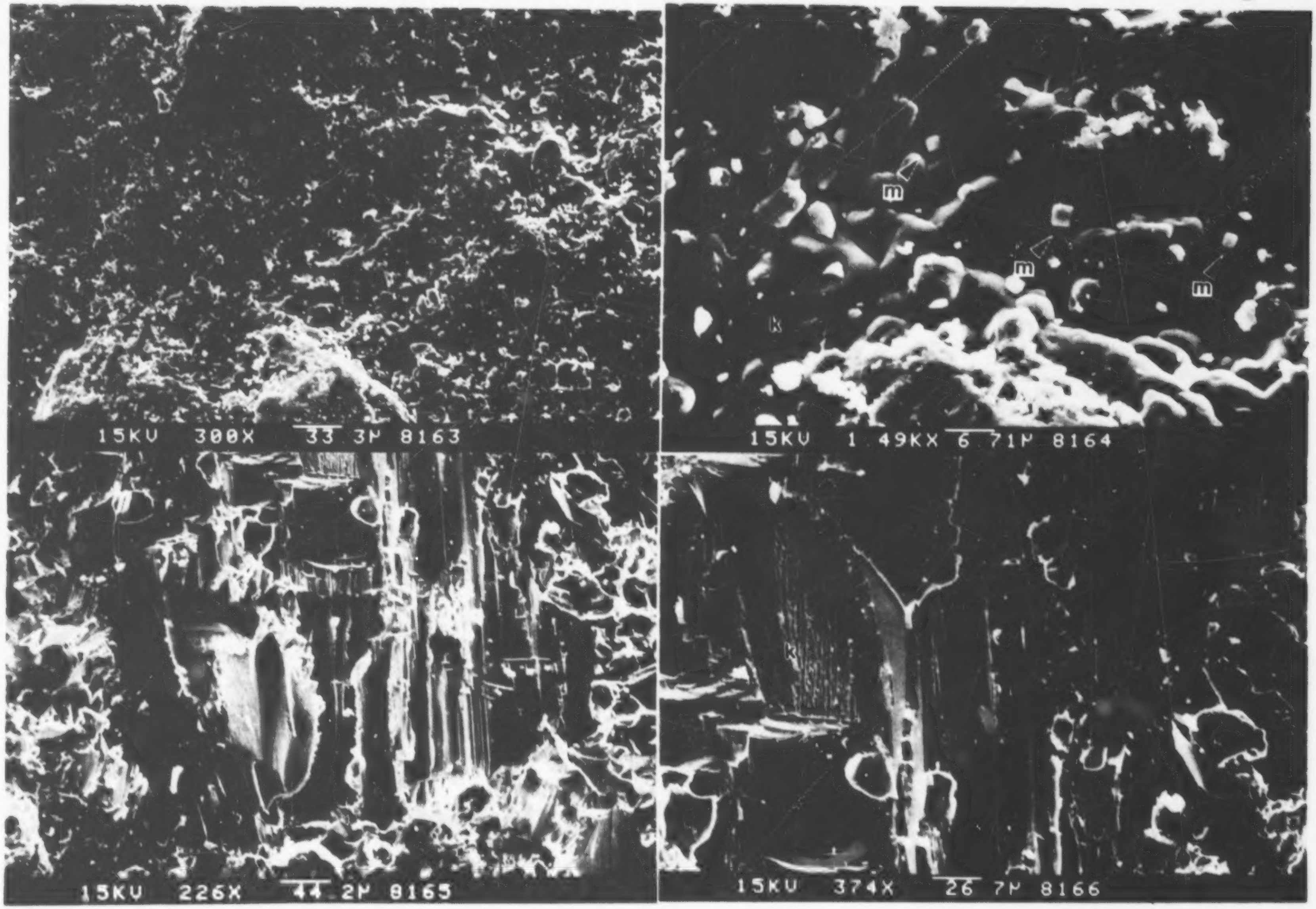
AS 54092
Figure Plate 3
Compacted Granular -8+10 Degrad


15KU 684X 14 6P 8076
15KU 2 60KX 3 85P 8077
m
m
k
k
n
n
n
15KU 2 59KX 3 86P 8078
n
LT= 60 SECS
8078 54092-2 FUZZPTT
COUNTS
ENERGY KeV
0.000
1.000
2.000
3.000
4.000
5.000
6.000
7.000
8.000
9.000
10.000

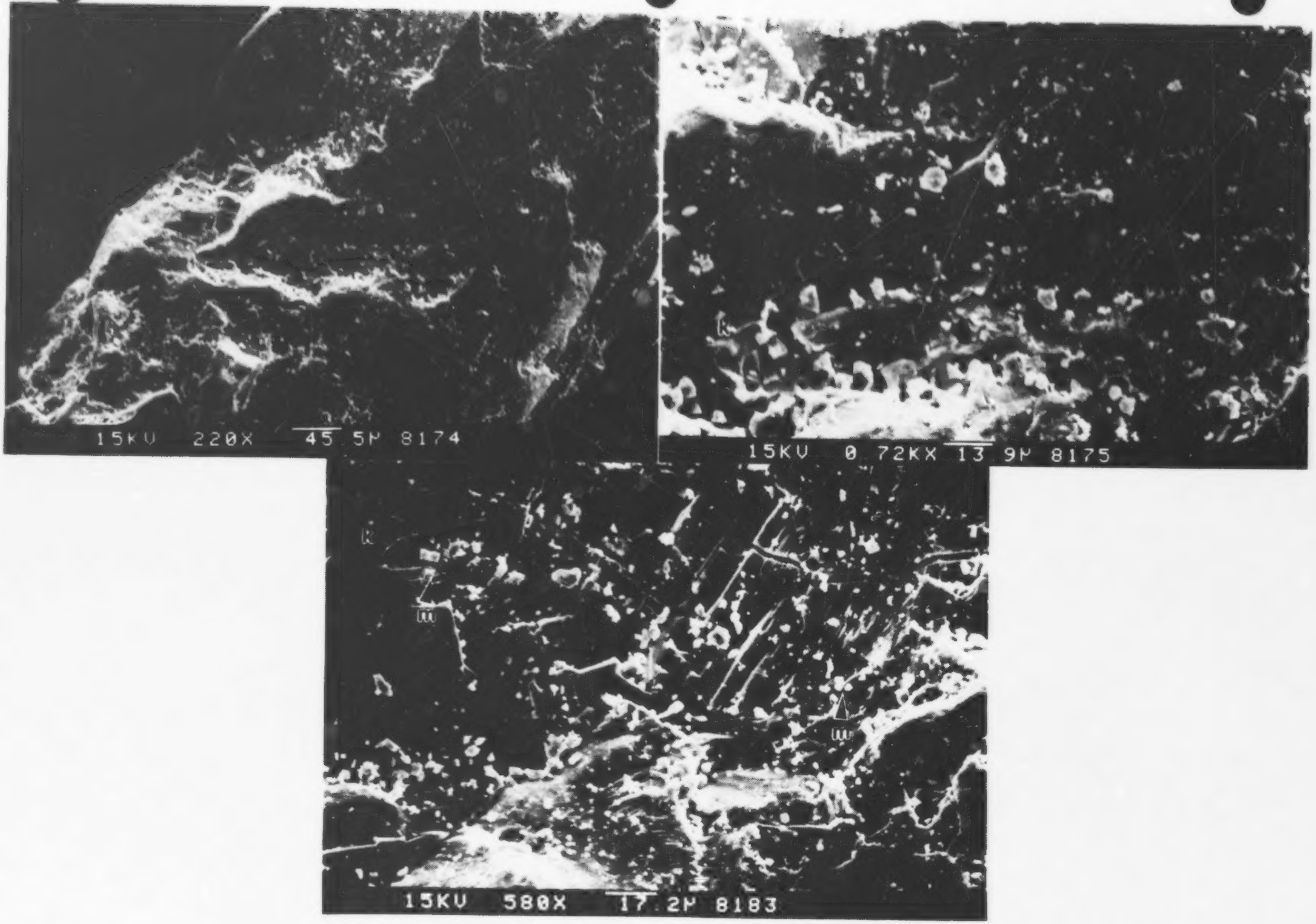
AS 54092
Figure Plate 5
Compacted Granular -8+10 Degrad


15KV 228X 43.9µ 8160
15KV 0.75KX 13.3µ 8161
15KV 1.51KX 6.62µ 8162

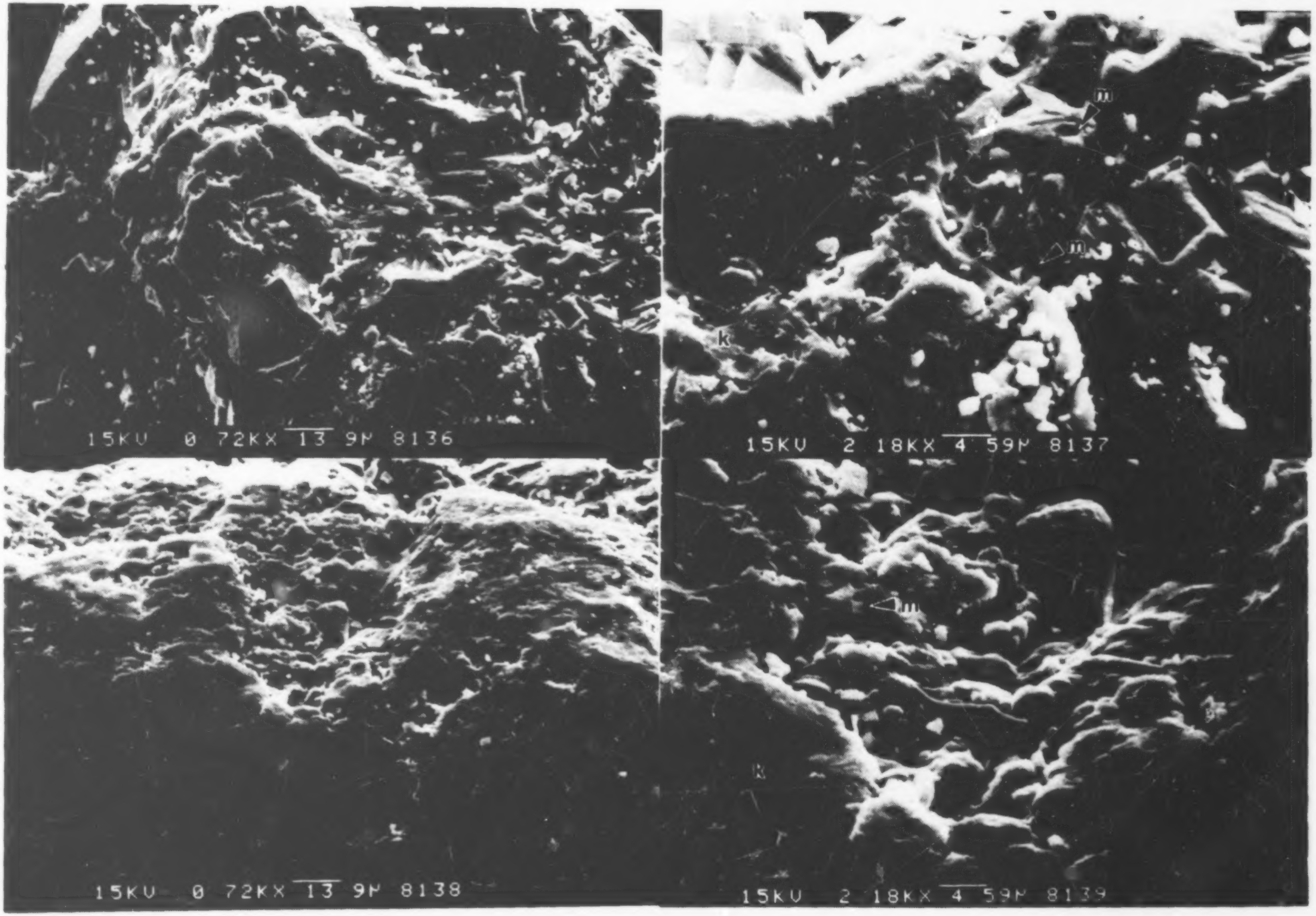
AS 54092
Figure Plate 30
Compacted Granular -10+14


15KV 300X 33 3P 8163
15KV 1 49KX 6 71P 8164
m
m
m
k
15KV 226X 44 2P 8165
15KV 374X 26 7P 8166
k

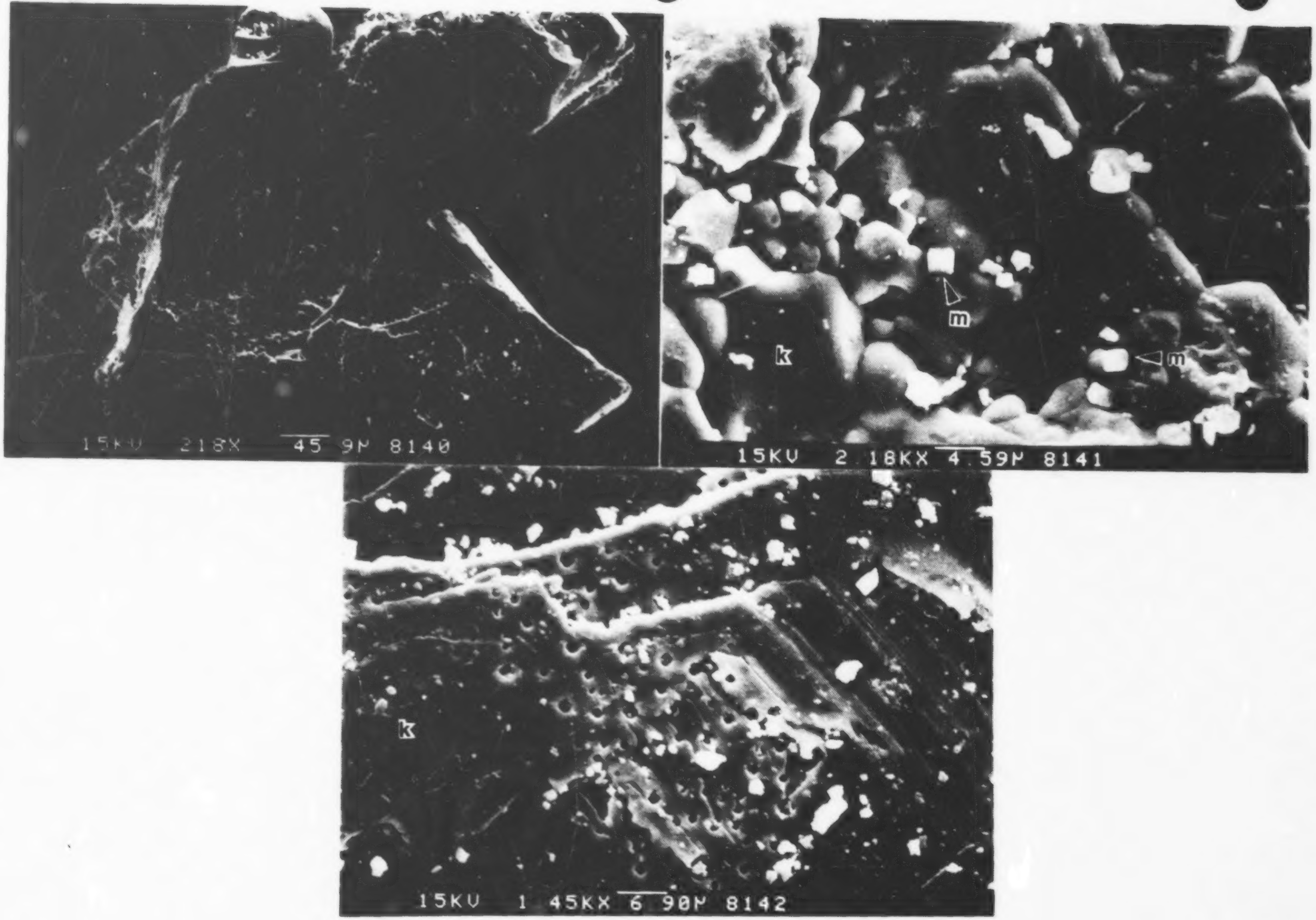
AS 54092
Figure Plate 31
Compacted Granular -10+14


15KV 220X 45 5P 8174
15KV 0 72KX 13 9P 8175
k
k
w
w
15KV 580X 17 2P 8183

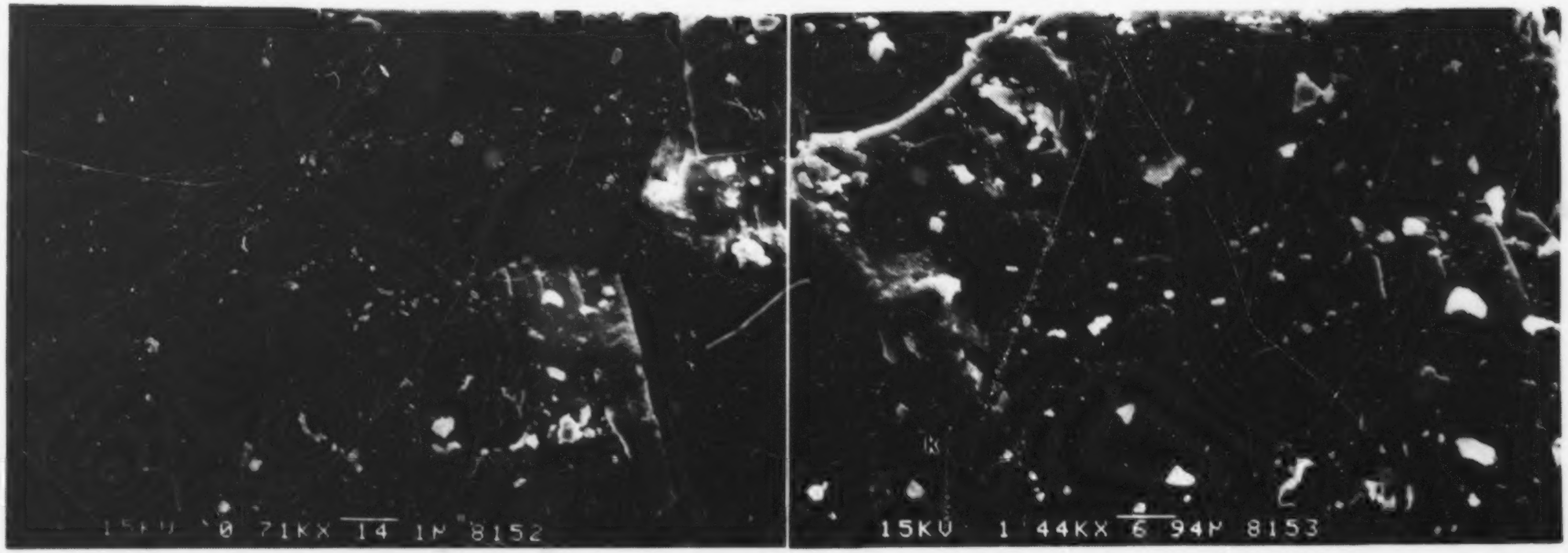
AS 54092
Figure Plate 34
Compacted Granular -35+48


15KV 0 72KX 13 9μ 8136
m
m
k
15KV 2 18KX 4 59μ 8137
15KV 0 72KX 13 9μ 8138
m
k
15KV 2 18KX 4 59μ 8139

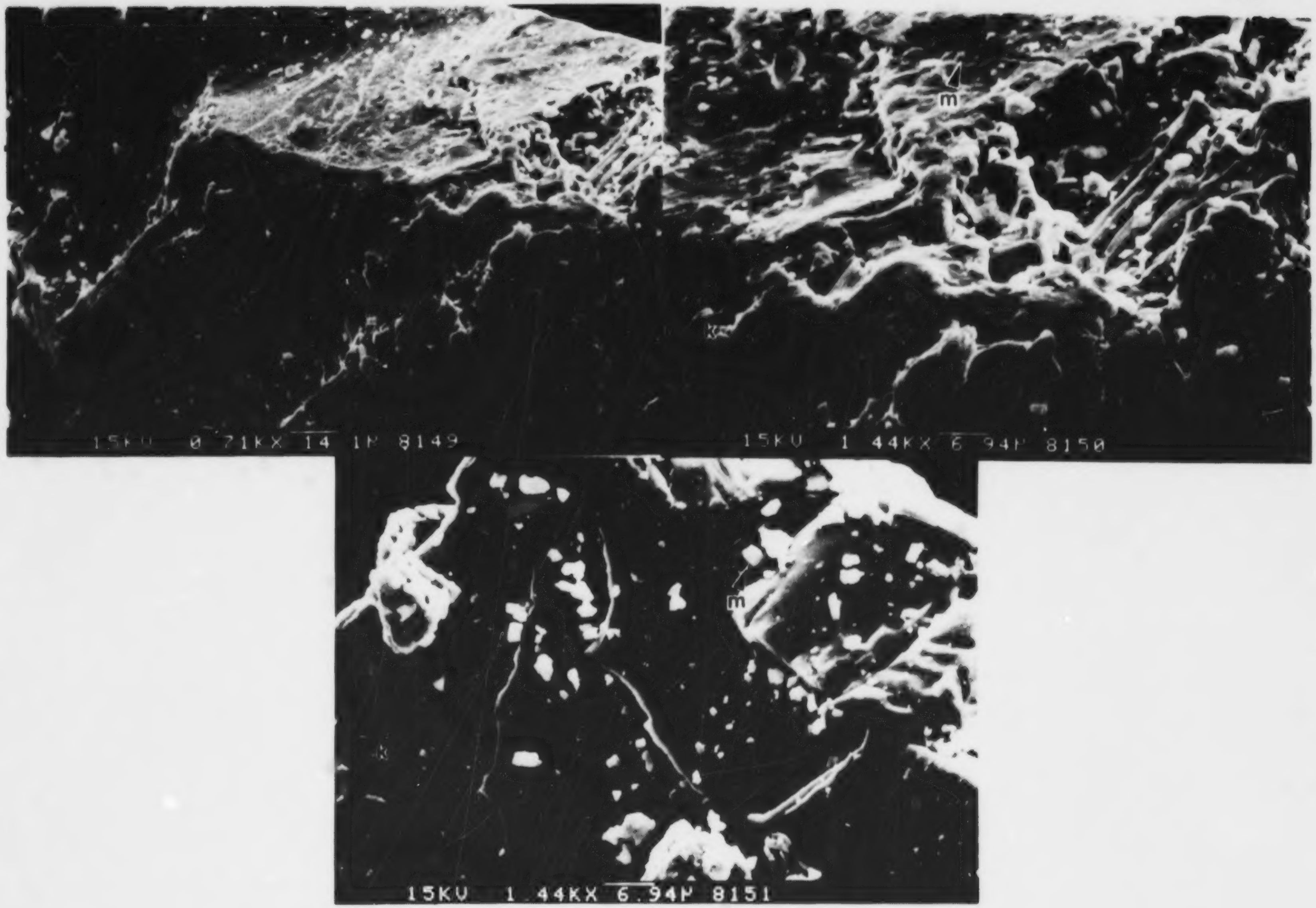
AS 54092
Figure Plate 23
Compacted Granular -65+100


15KV 218X 45 9µ 8140
15KV 2 18KX 4 59µ 8141
k
m
m
15KV 1 45KX 6 90µ 8142
k

AS 54092
Figure Plate 22
Compacted Granular -65+100

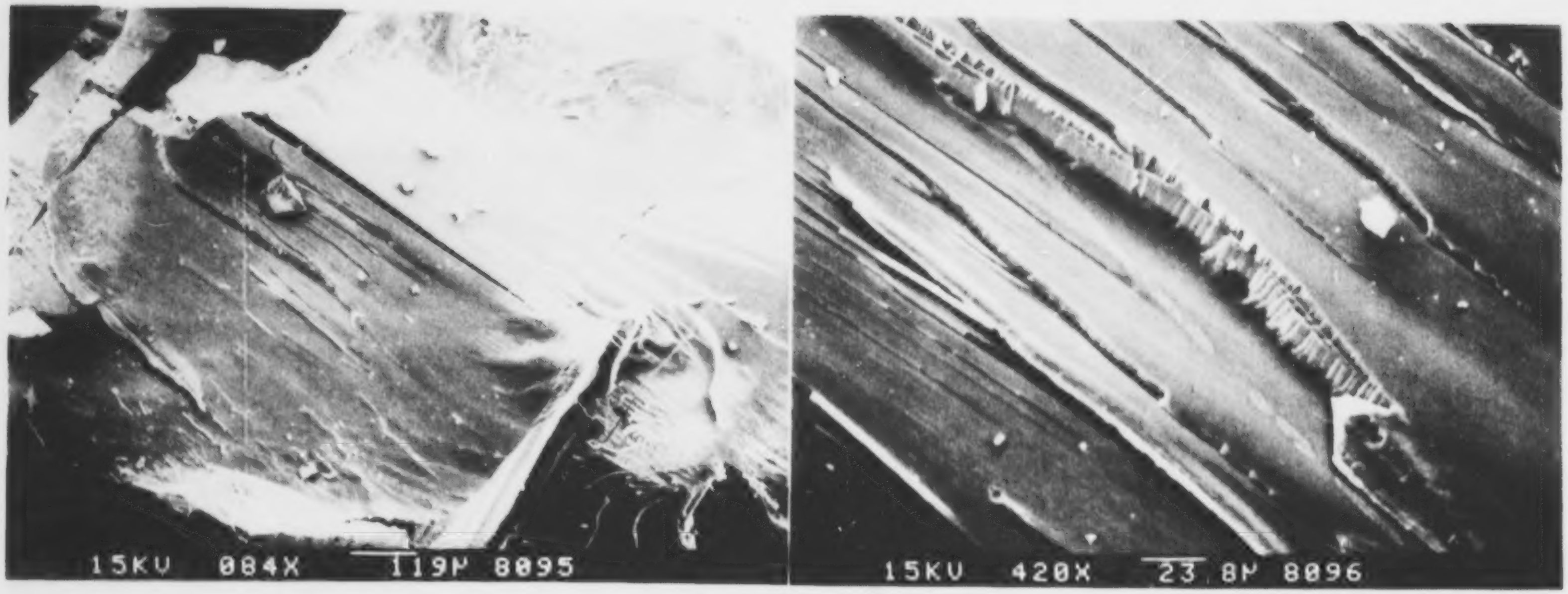

15KV 0 71KX 14 1P 8152
15KV 1 44KX 6 94P 8153

AS 54092
Figure Plate 27
Compacted Granular -100

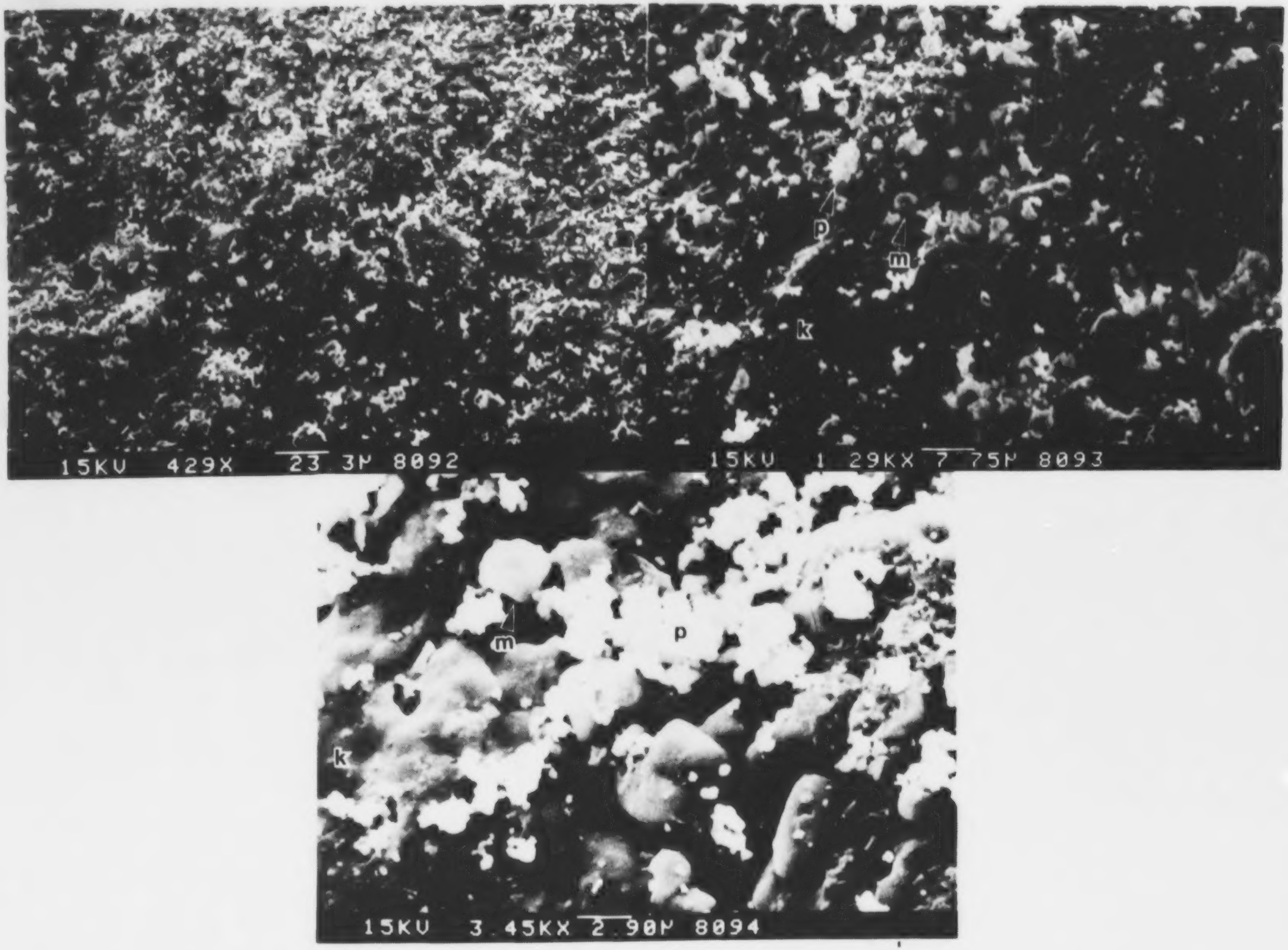

15KV 0.71KX 14.1µ 8149
m
15KV 1.44KX 6.94µ 8150
m
15KV 1.44KX 6.94µ 8151

AS 54092
Figure Plate 26
Compacted Granular -100

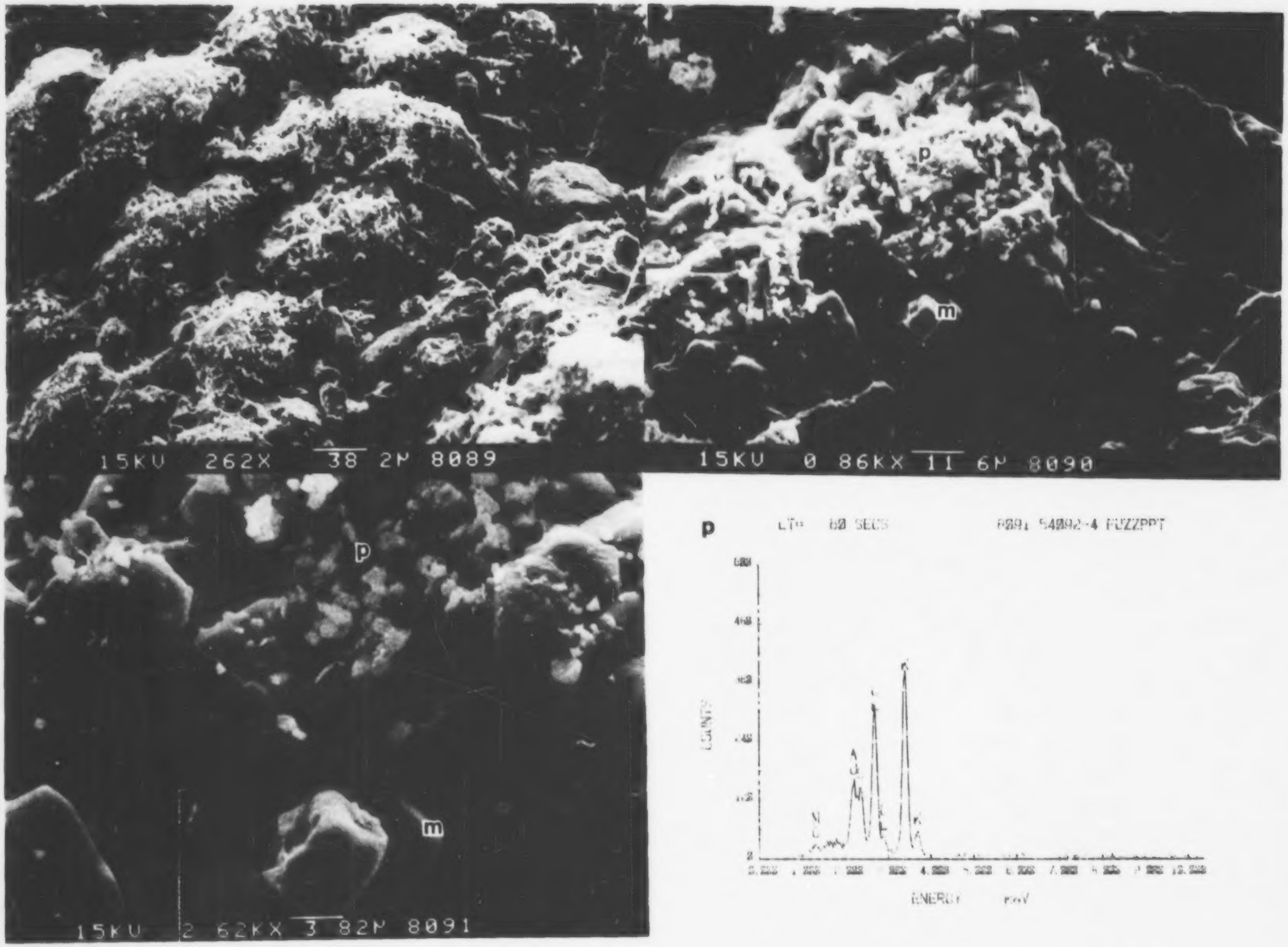
PLATES


15KV 084X 119μ 8095
15KV 420X 23.8μ 8096

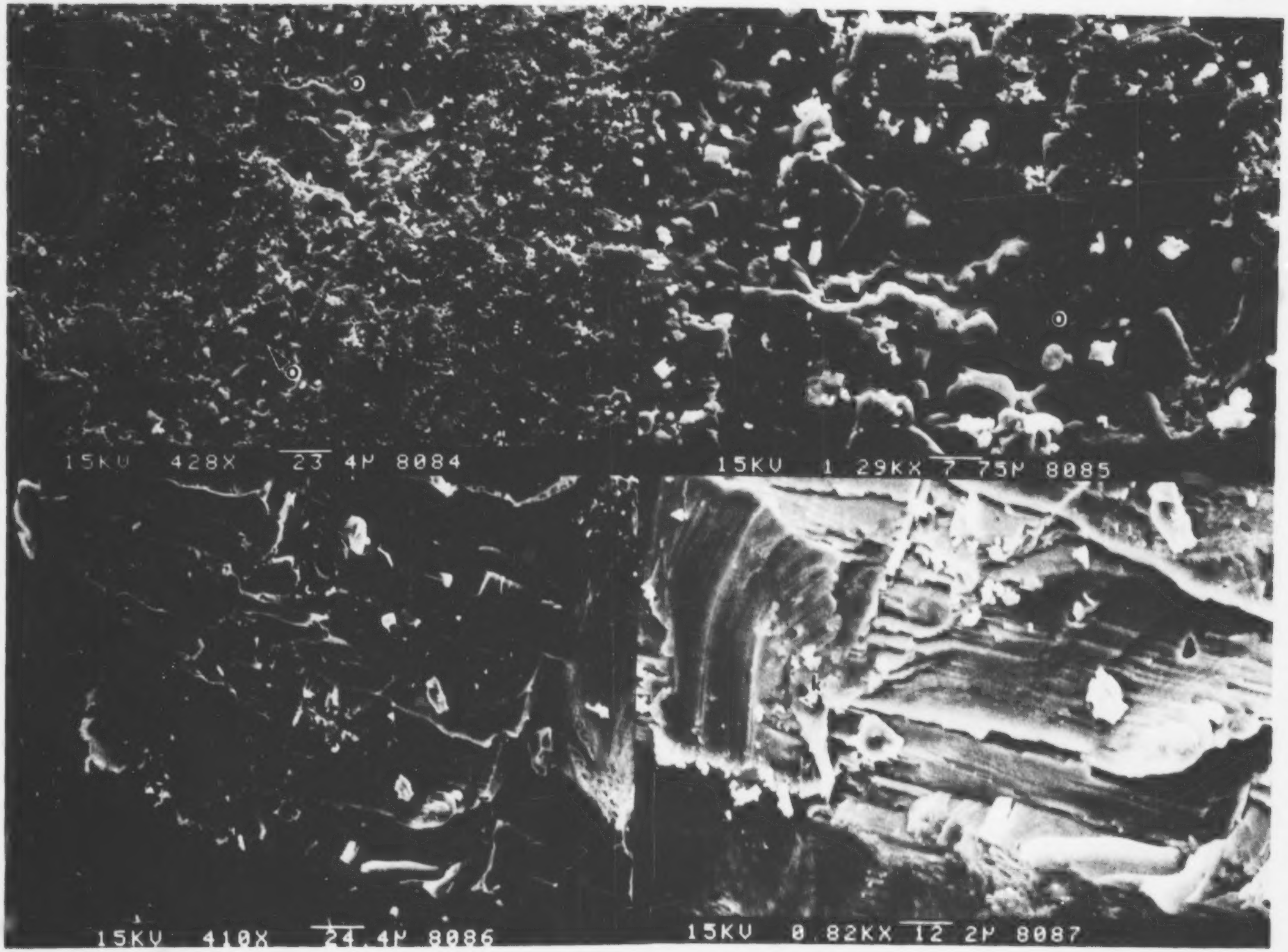
AS 54092
Figure Plate 11
Lanigan Natural Coarse -8+10 Not degrad


15KV 429X 23 3P 8092
15KV 1 29KX 7 75P 8093
p
m
k
15KV 3 45KX 2 90P 8094
m
p
k

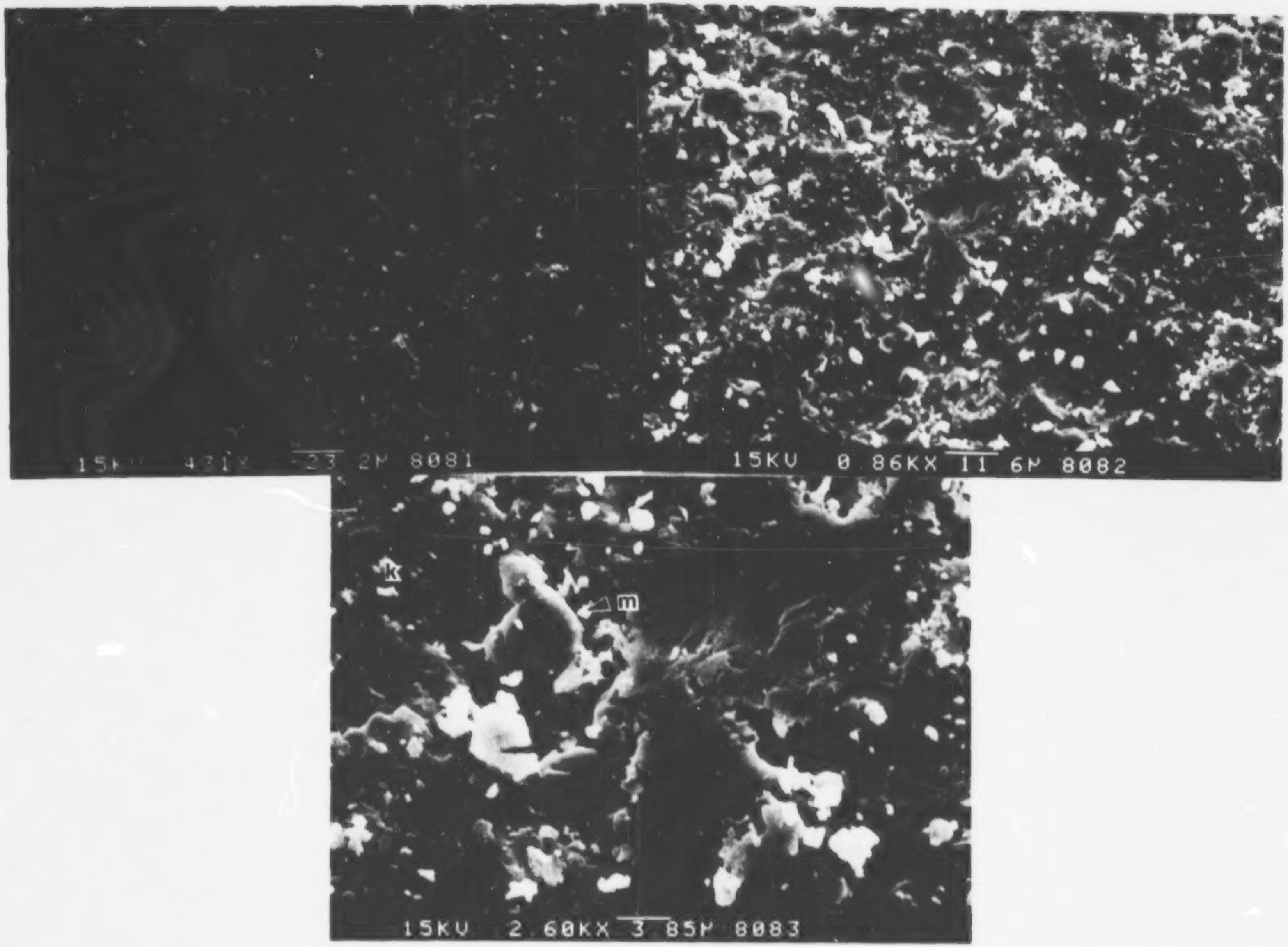
AS 54092
Figure Plate 10
Lanigan Natural Coarse -8+10 Not degrad


15KV 262X 38 2P 8089
15KV 0 86KX 11 6P 8090
p
m
p
m
15KV 2 62KX 3 82P 8091
p
LT= 60 SECS
8091 54092-4 FUZZPPT
COUNTS
ENERGY keV
0
4.000
10.000

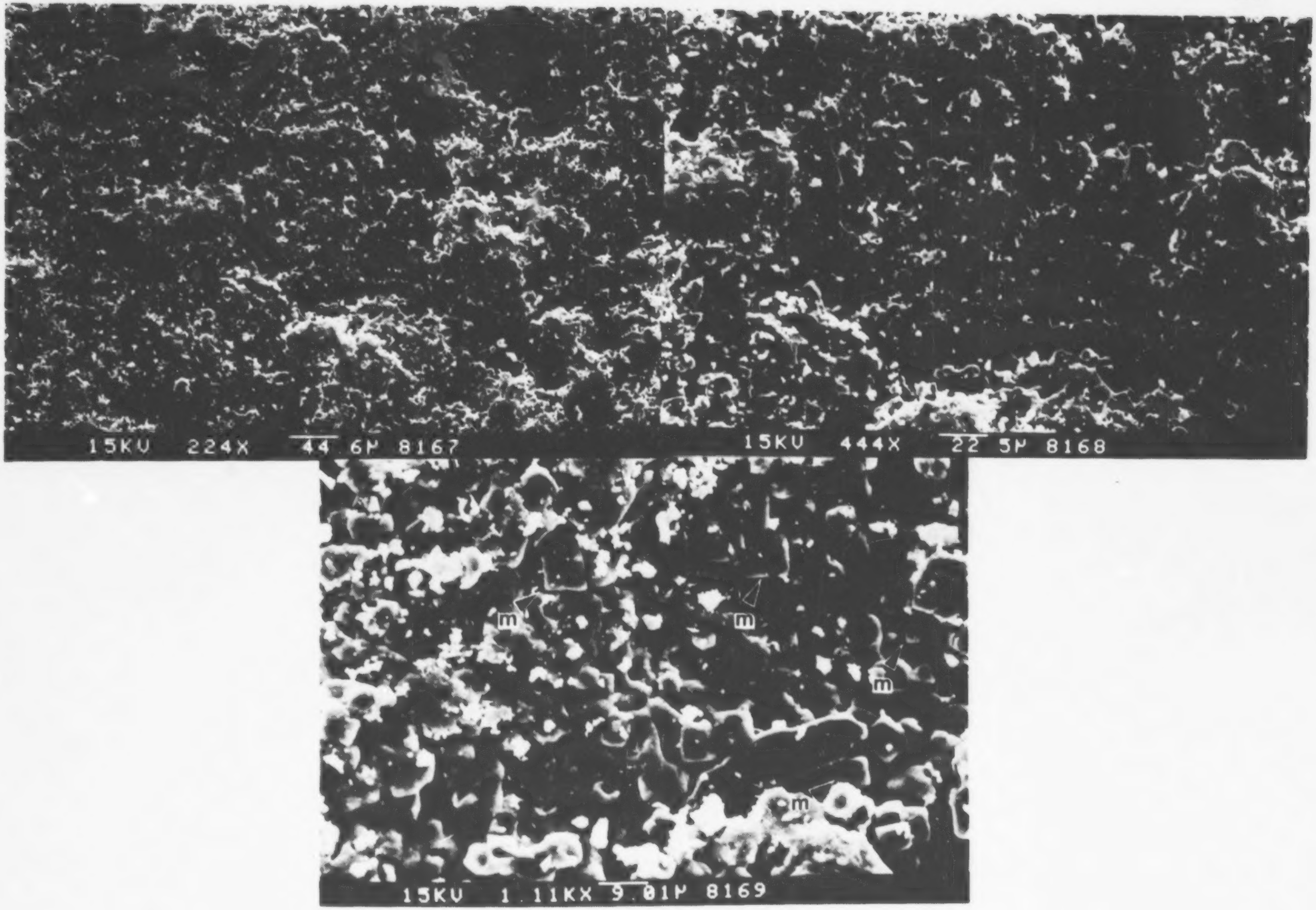
AS 54092
Figure Plate 9
Lanigan Natural Coarse -8+10 Not degrad


15KU 428X 23 4µ 8084
15KU 1 29KX 7 75µ 8085
15KU 410X 24 4µ 8086
15KU 0 82KX 12 2µ 8087

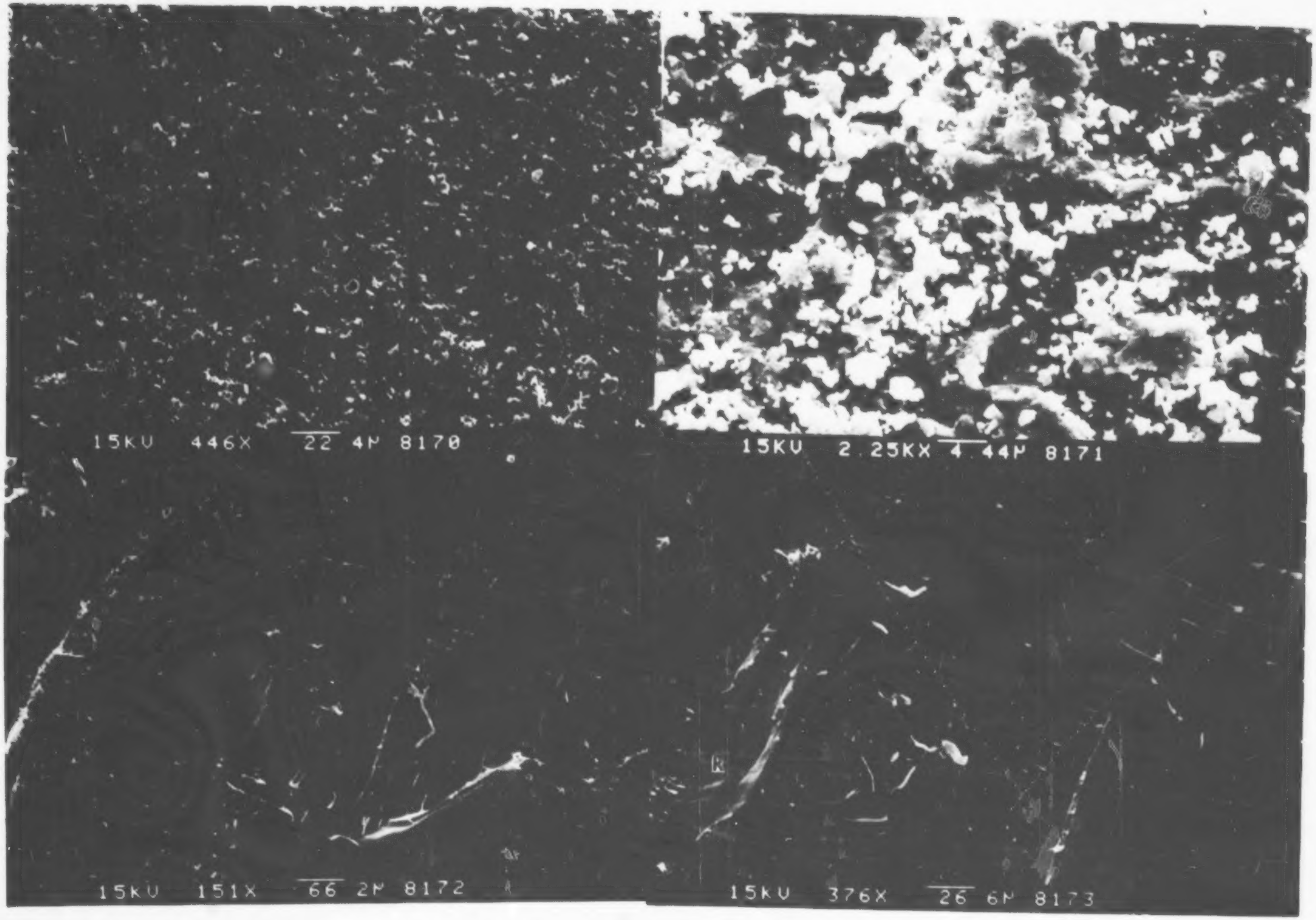
AS 54092
Figure PLate 8
Lanigan Natural Coarse -8+10 Degrad


15KV 431X 23 2µ 8081
15KV 0 86KX 11 6µ 8082
k
m
15KV 2 60KX 3 85µ 8083

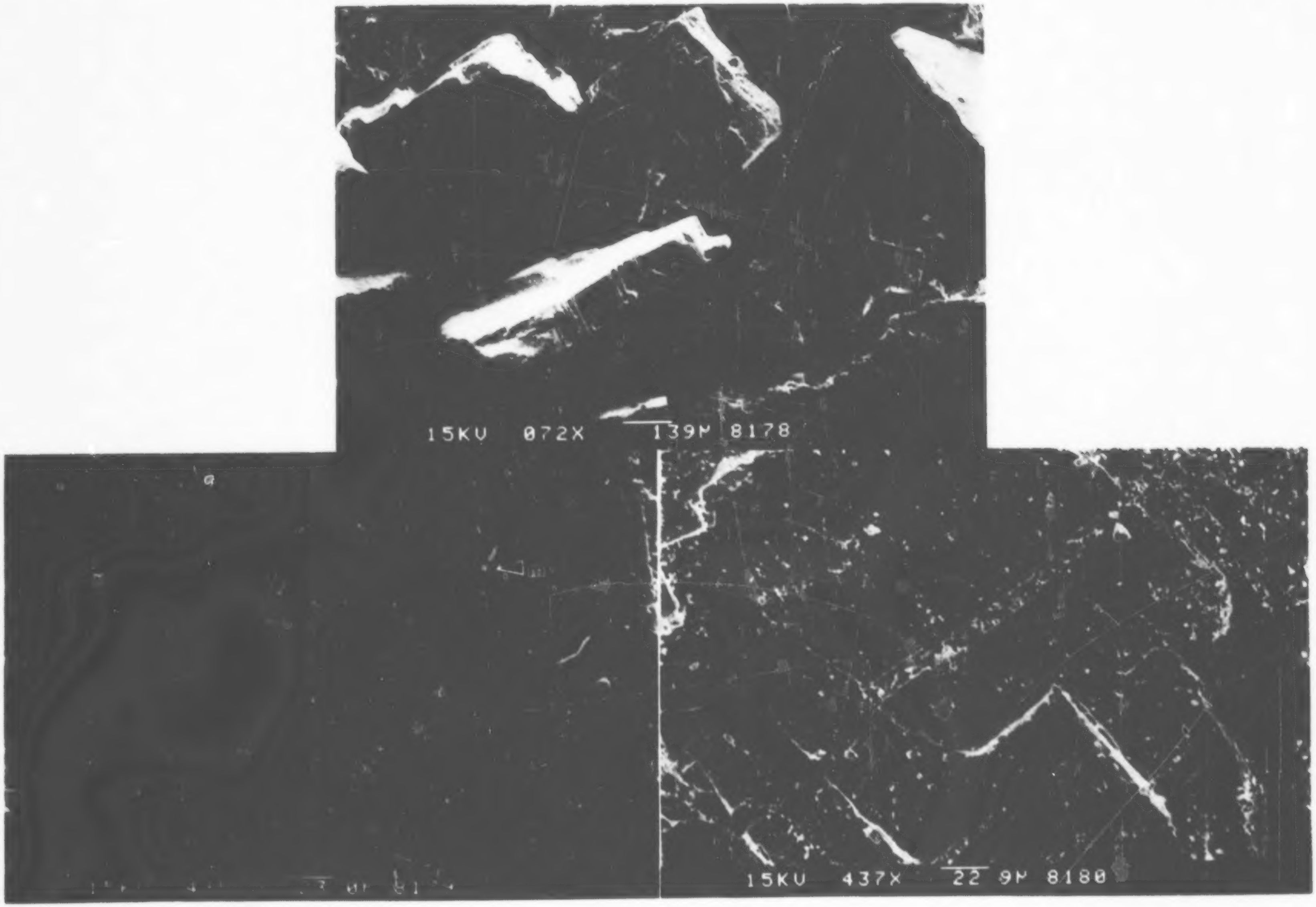
AS 54092
Figure Plate 7
Lanigan Natural Coarse -8+10 Degrad


15KV 224X 44 6P 8167
15KV 444X 22 5P 8168
m
m
m
m
15KV 1 11KX 9 01P 8169

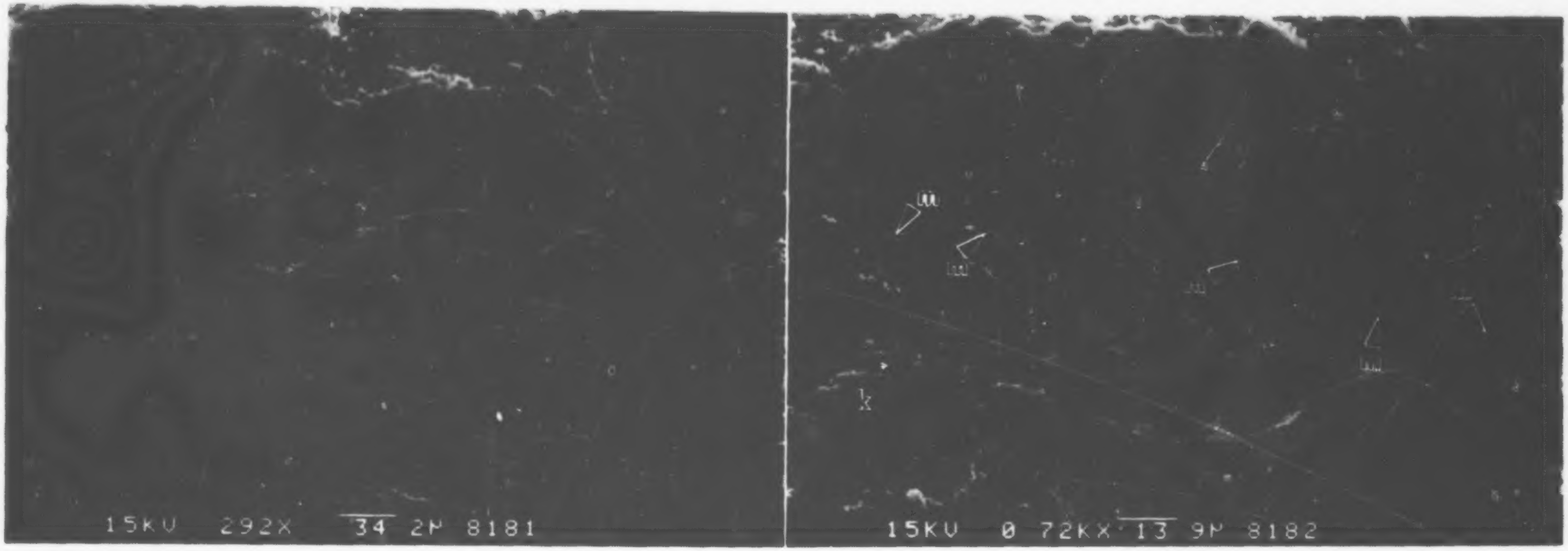
AS 54092
Figure Plate 32
Lanigan Natural Coarse -10+14


15KU 446X 22 4µ 8170
15KU 2.25KX 4.44µ 8171
15KU 151X 66 2µ 8172
15KU 376X 26 6µ 8173

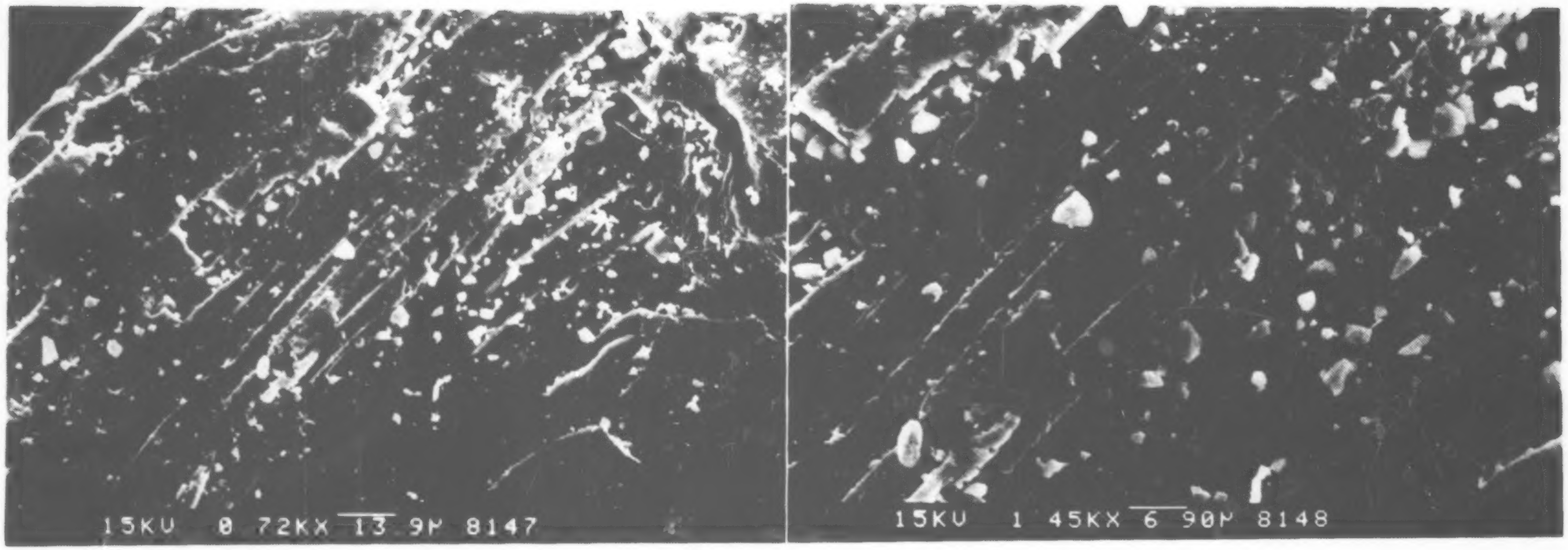
AS 54092
Figure Plate 33
Lanigan Natural Coarse -10+14


15KV 072X 139µ 8178
15KV 437X 22 9µ 8180

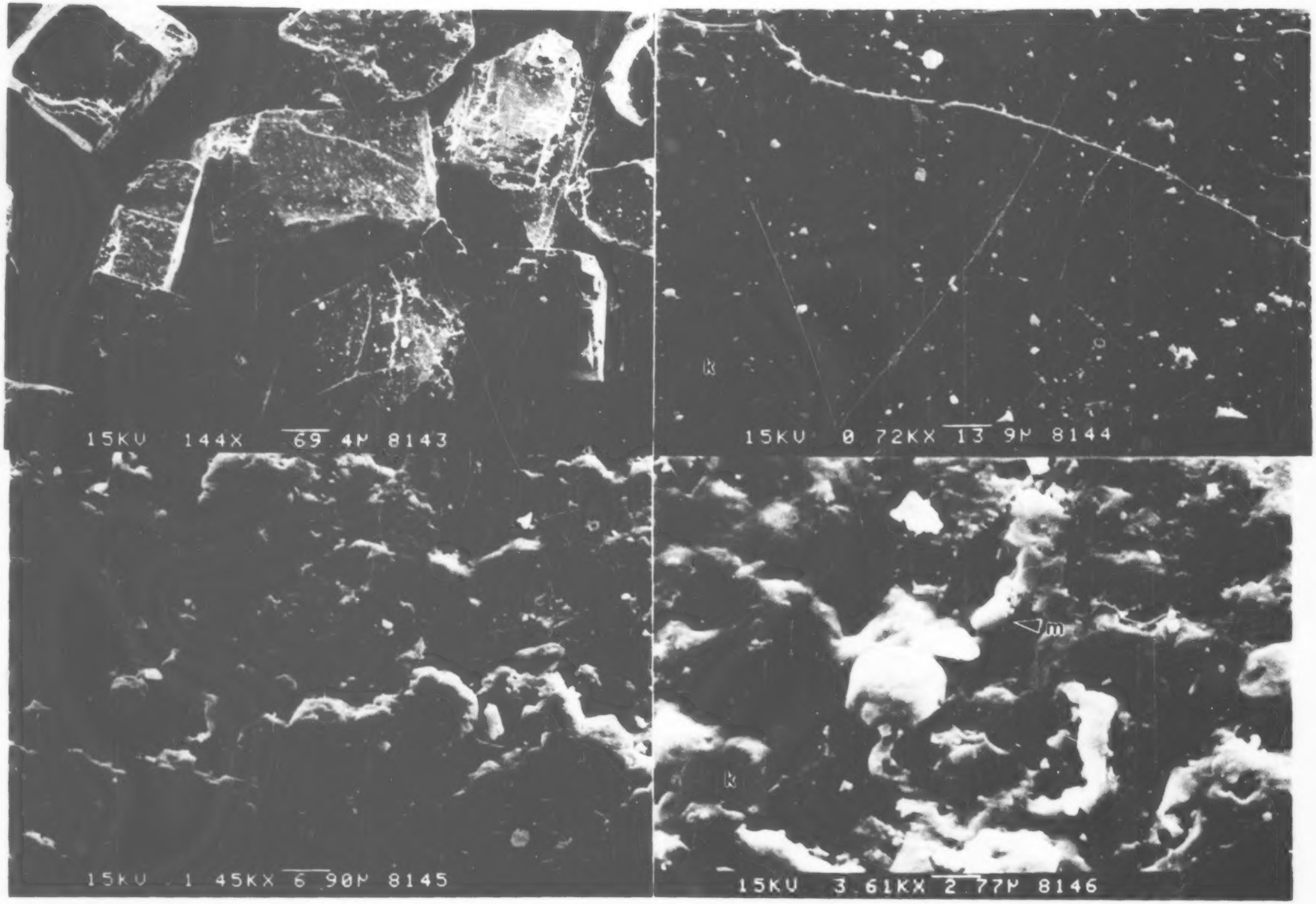
AS 54092
Figure Plate 36
Lanigan Natural Coarse -35+48


15KV 292X 34 2µ 8181
15KV 0 72KX 13 9µ 8182
k

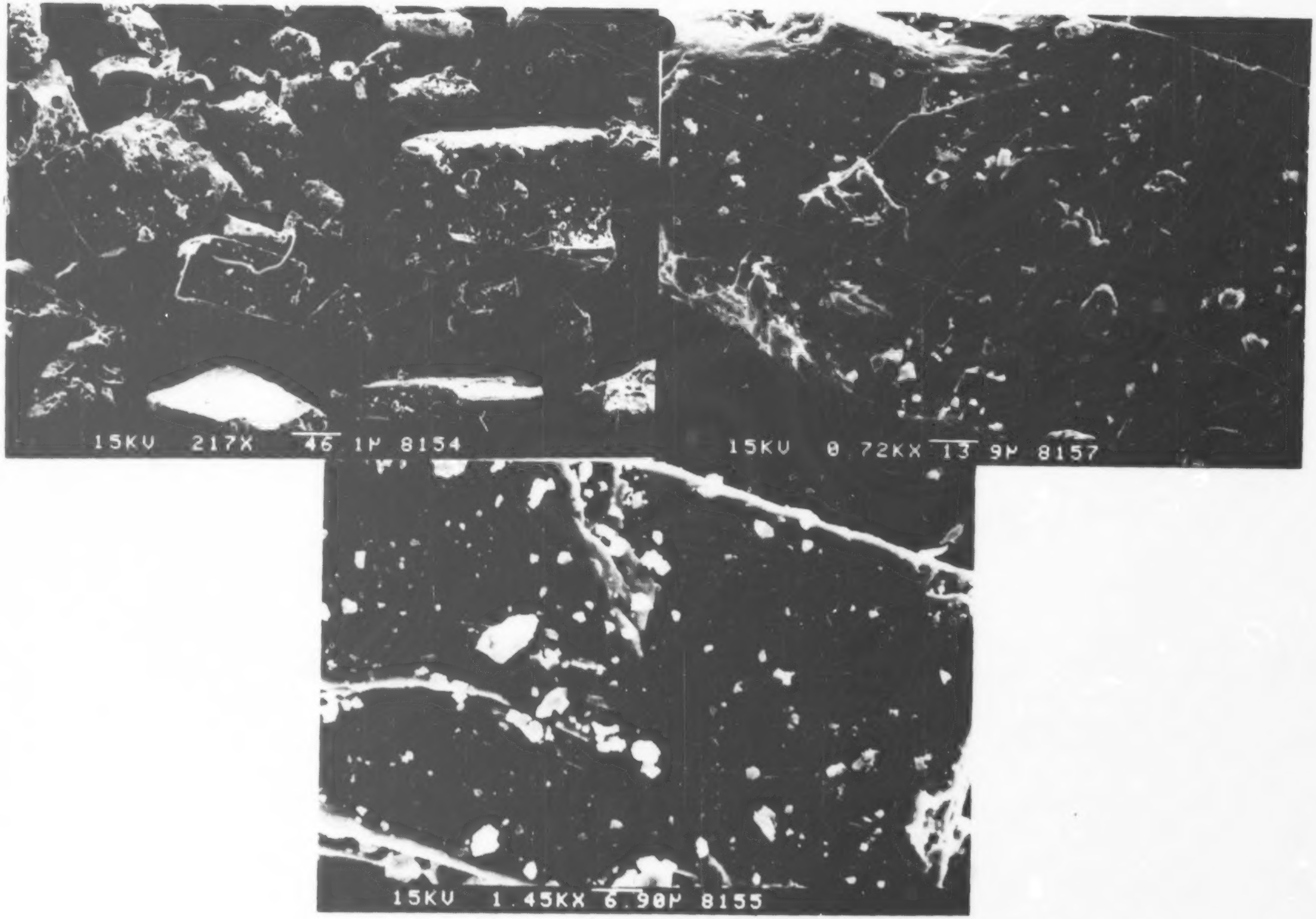
AS 54092
Figure Plate 37
Lanigan Natural Coarse -35+48


15KU 0 72KX 13 9µ 8147
15KU 1 45KX 6 90µ 8148

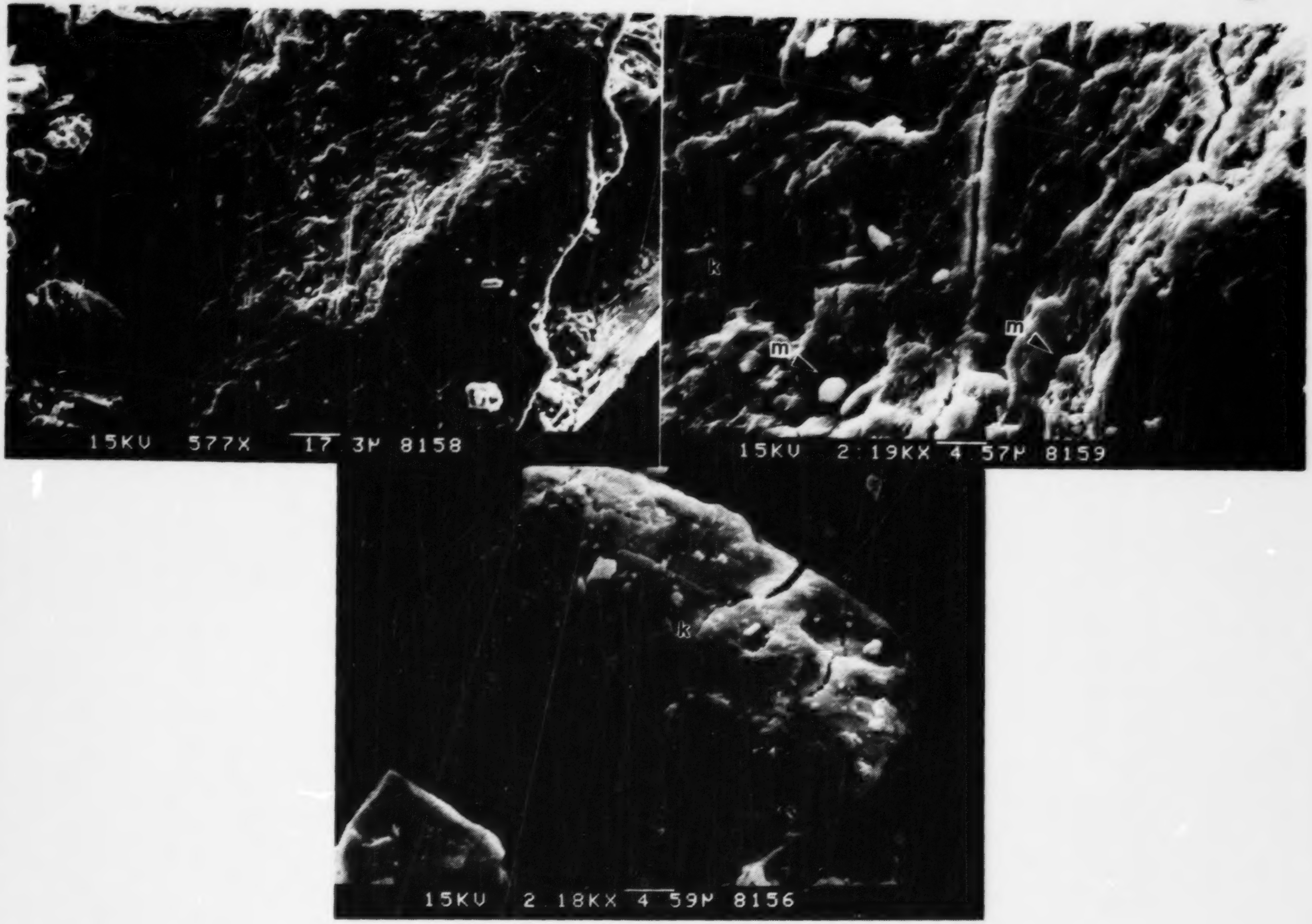
AS 54092
Figure Plate 25
Lanigan Natural Coarse -65+100


15KV 144X 69 4P 8143
k
15KV 0 72KX 13 9P 8144
15KV 1 45KX 6 90P 8145
m
k
15KV 3 61KX 2 77P 8146

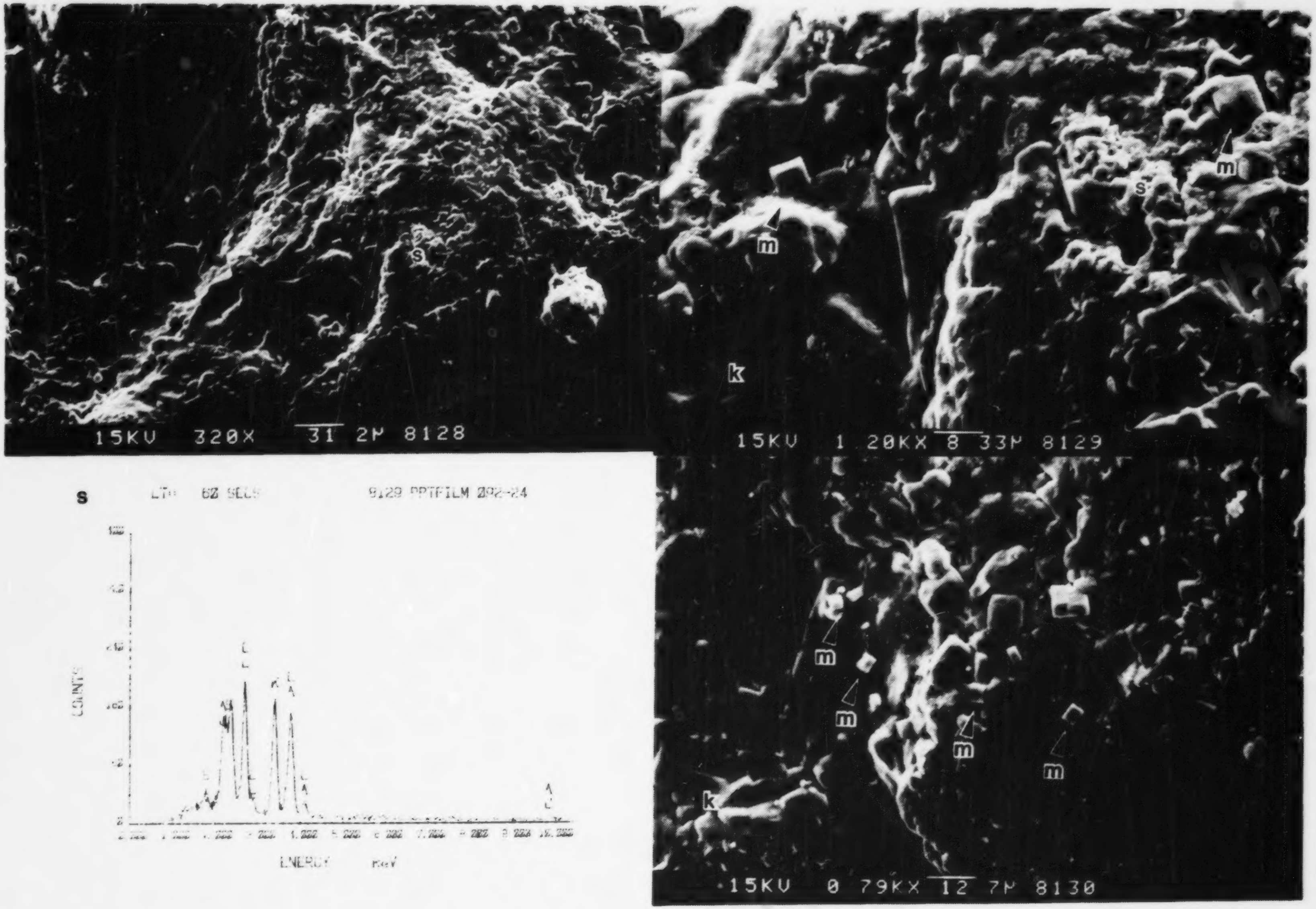
AS 54092
Figure Plate 24
Lanigan Natural Coarse -65+100


15KV 217X 46.1μ 8154
15KV 0.72KX 13.9μ 8157
15KV 1.45KX 6.90μ 8155

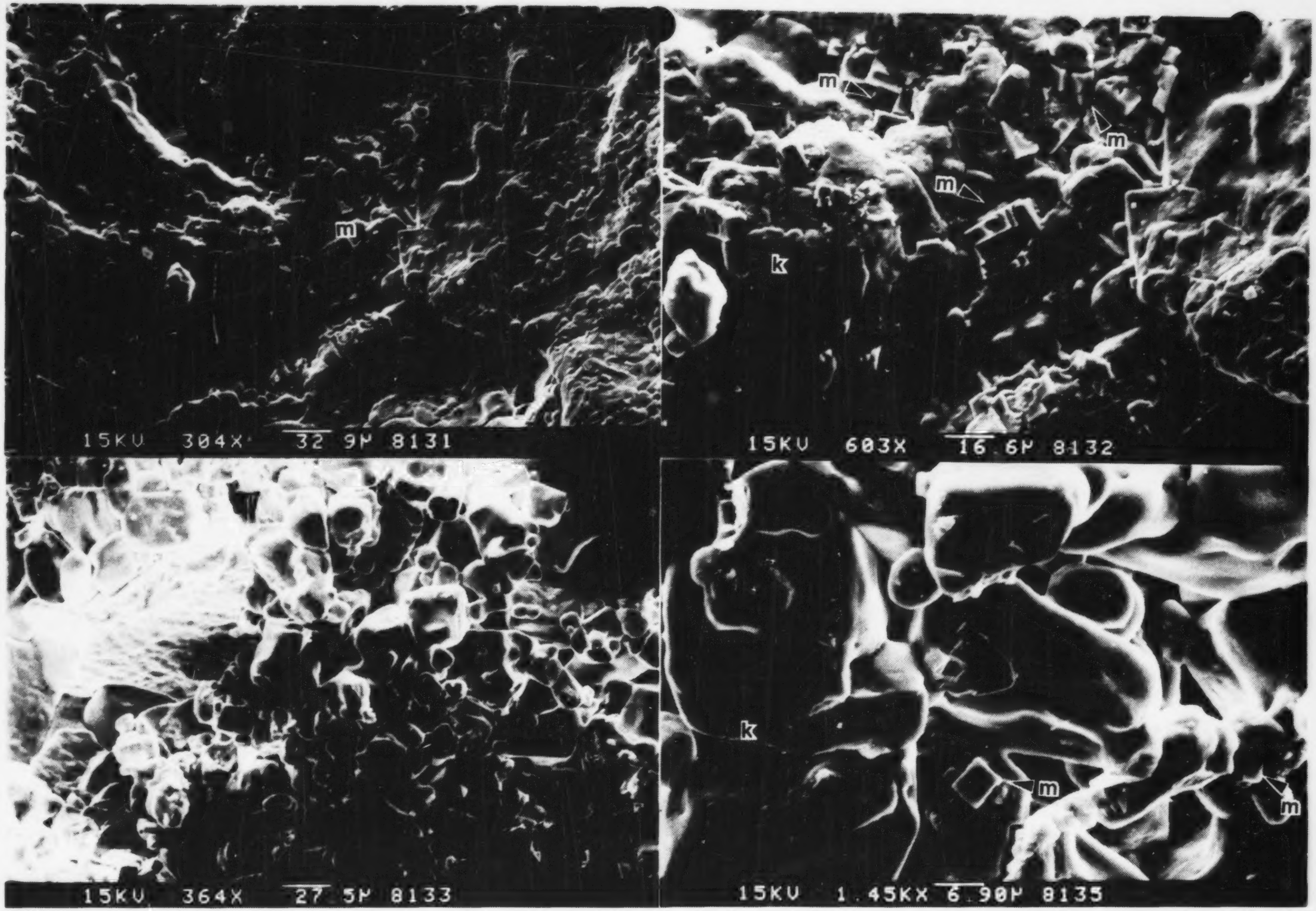
AS 54092
Figure Plate 28
Lanigan Natural Coarse -100


15KV 577X 17 3µ 8158
k
m
m
15KV 2 19KX 4 57µ 8159
k
15KV 2 18KX 4 59µ 8156

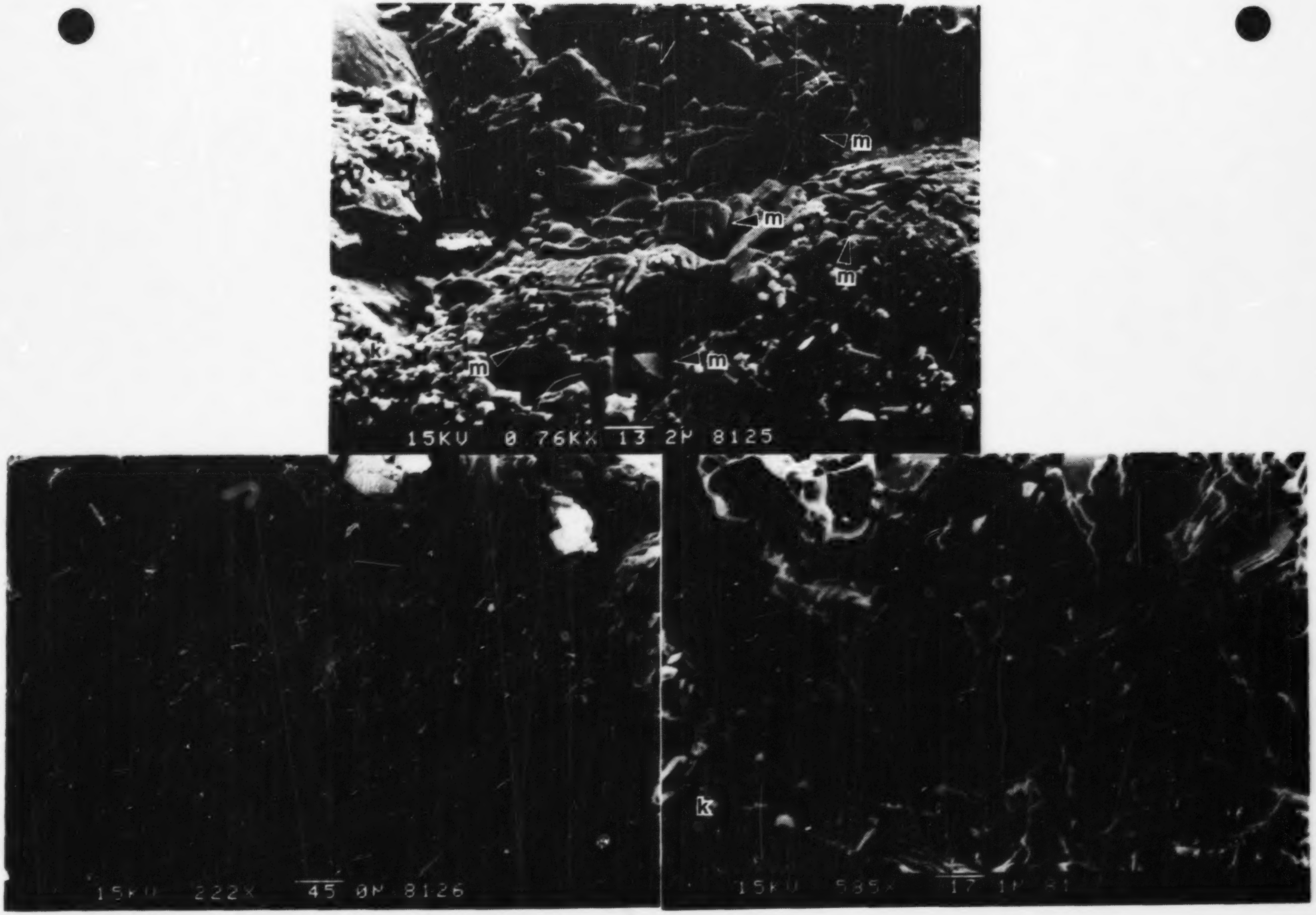
AS 54092
Figure Plate 29
Lanigan Natural Coarse -100


15KV 320X 31 2P 8128
s
15KV 1 20KX 8 33P 8129
m
m
s
k
15KV 0 79KX 12 7P 8130
m
m
m
m
m
k
s
LT= 60 SECS
8129 PPTFILM 092-24
COUNTS
ENERGY KeV
1.000
4.000
5.000
6.000
7.000
8.000
9.000
10.000

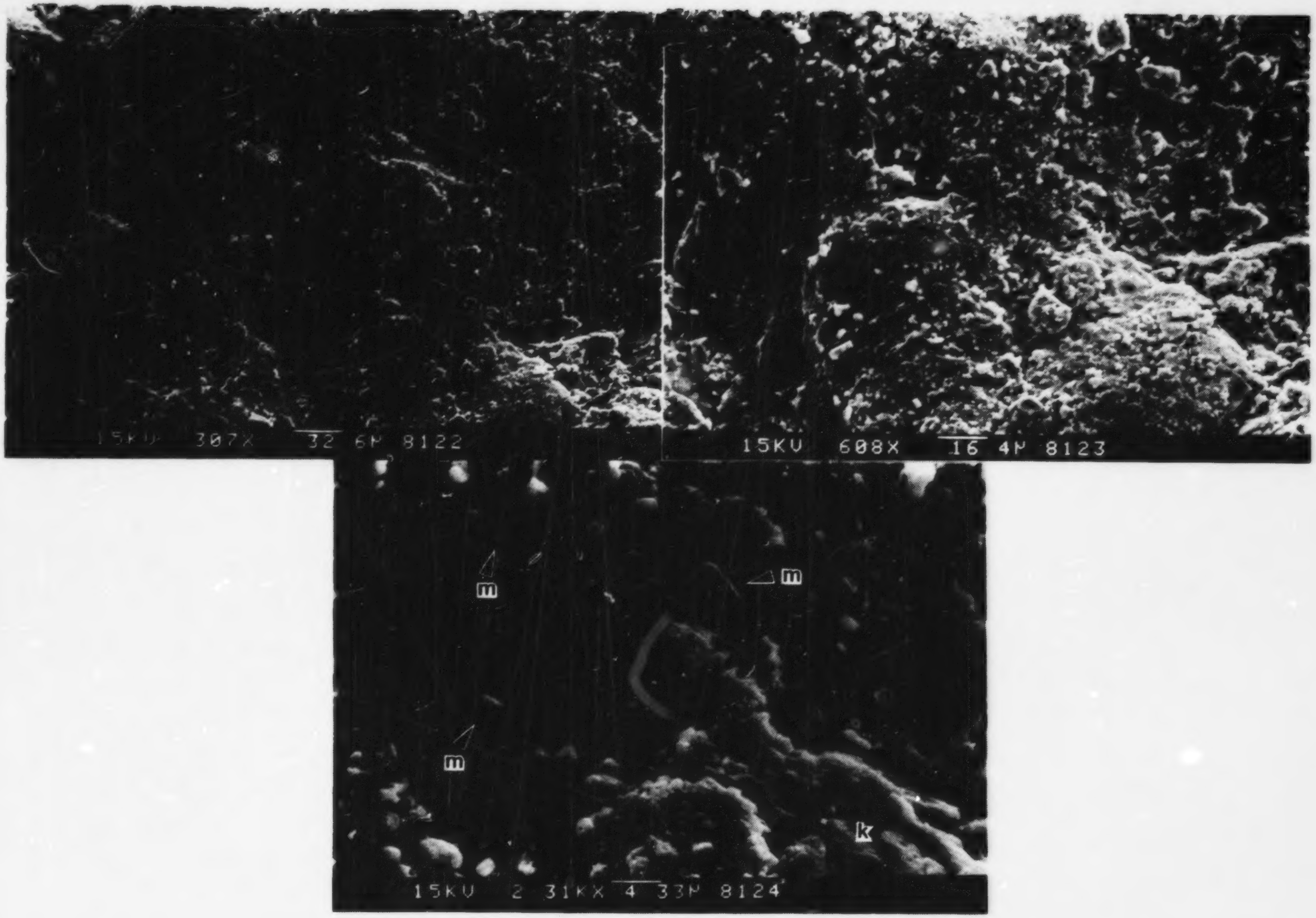
AS 54092
Figure Plate 20
No Treatment


m
15KV 304X 32 9P 8131
m
m
m
k
15KV 603X 16 6P 8132
15KV 364X 27 5P 8133
k
m
m
15KV 1 45KX 6 90P 8135

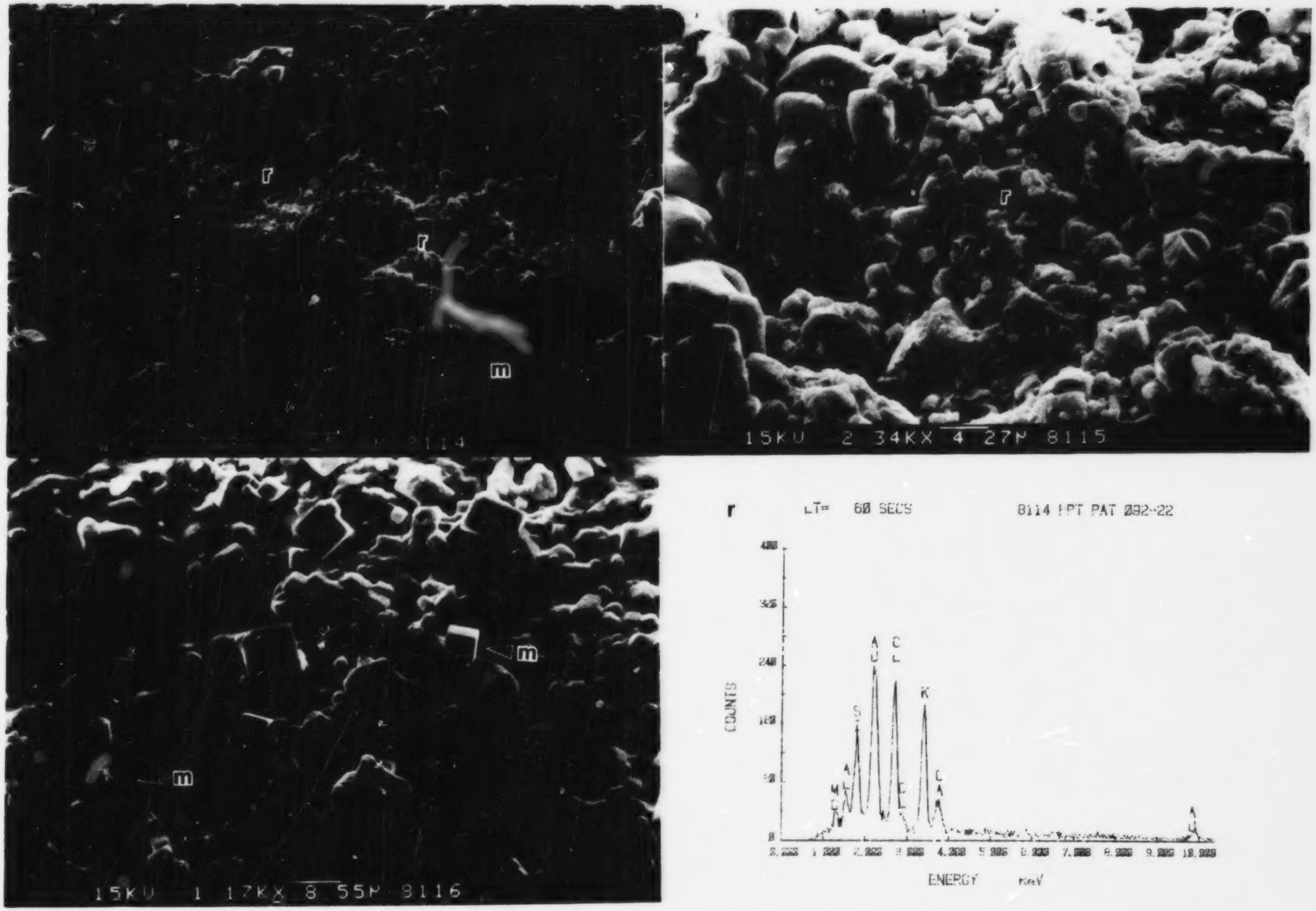
AS 54092
Figure Plate 21
No Treatment


m
m
m
m
m
m
k
15KV 0 76KX 13 2µ 8125
15KV 222X 45 0µ 8126
k
15KV 585X 17 1µ 81

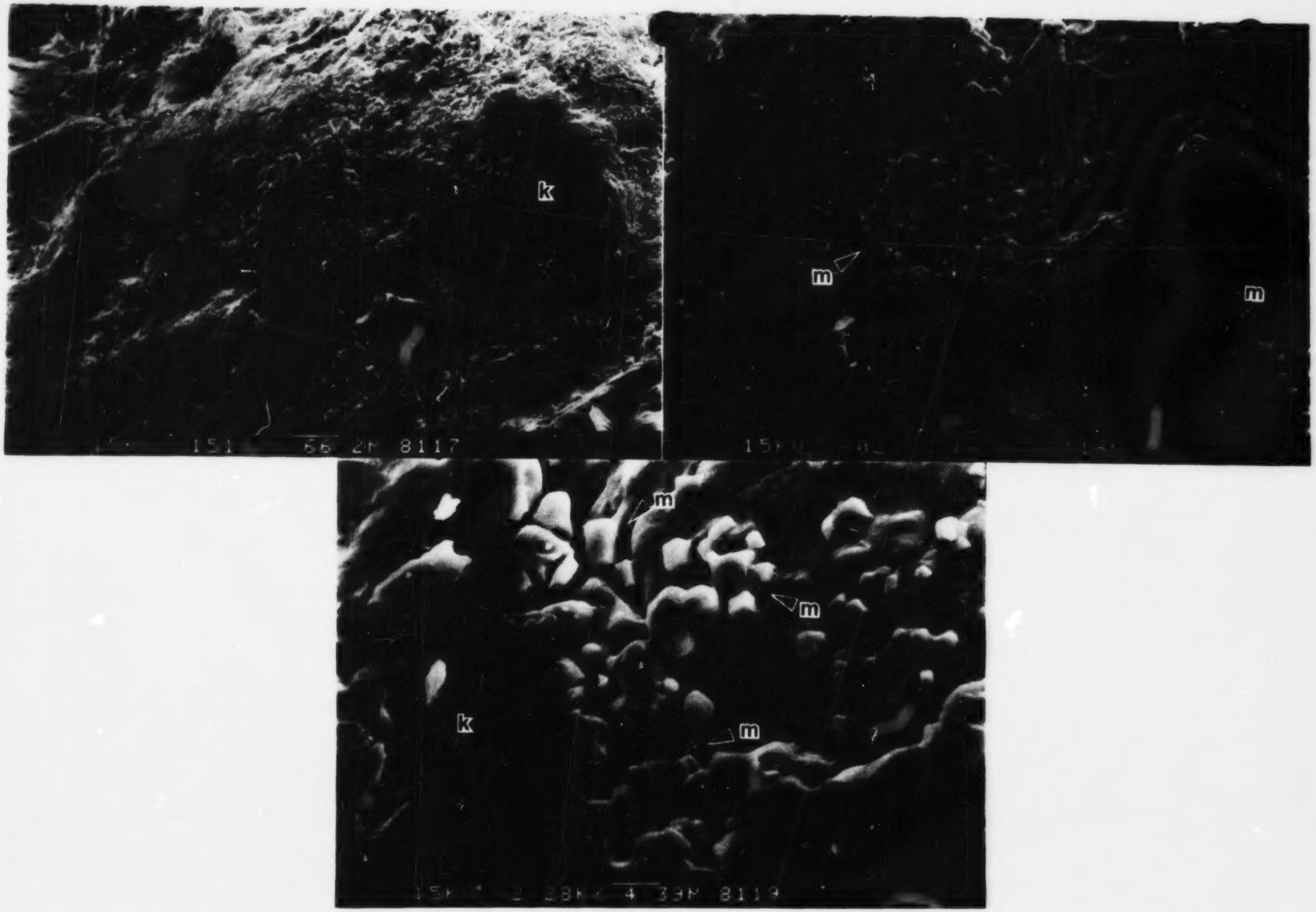
AS 54092
Figure Plate 19
Husky-Armac T


15KV 307X 32 6μ 8122
15KV 608X 16 4μ 8123
m
m
m
k
15KV 2 31KX 4 33μ 8124

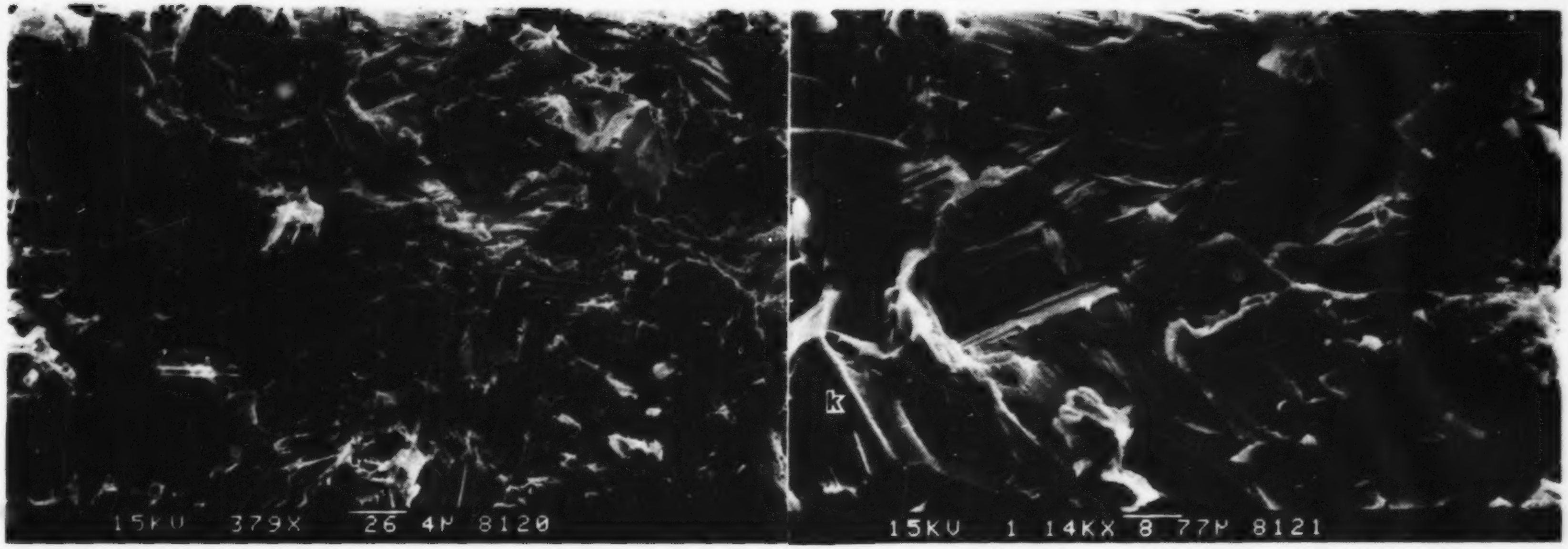
AS 54092
Figure Plate 18
Husky-Armac T


r
r
m
8114
r
15KU 2 34KX 4 27P 8115
m
m
15KU 1 17KX 8 55P 8116
r
LT= 60 SECS
8114 I PT PAT 092-22
COUNTS
400
320
240
160
80
0
M G
A L
S I
A U
C L
C L
K
C A
A U
0.000
1.000
2.000
3.000
4.000
5.000
6.000
7.000
8.000
9.000
10.000
ENERGY
KeV

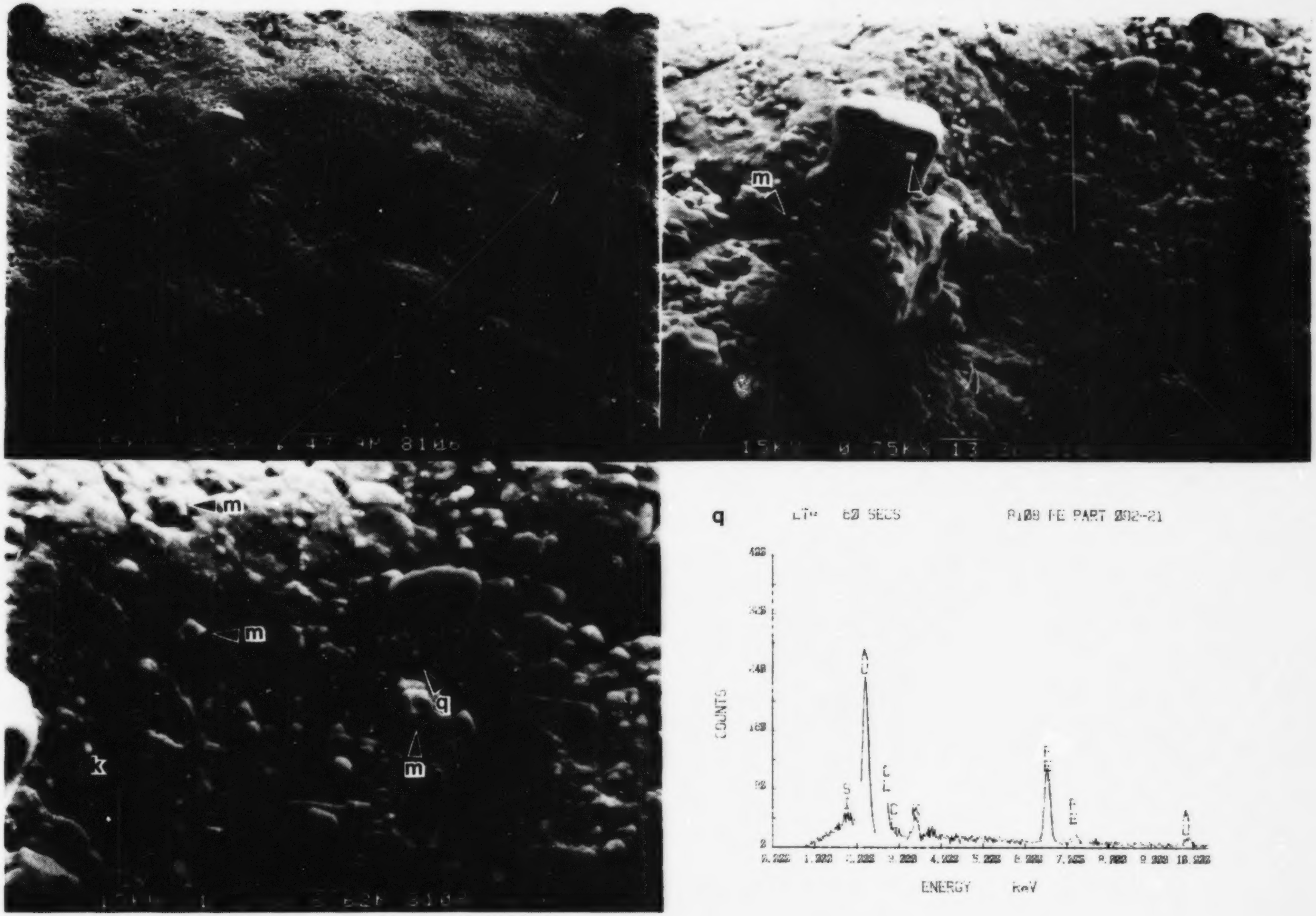
AS 54092
Figure Plate 15
Canola-Arosurf MG 102 W



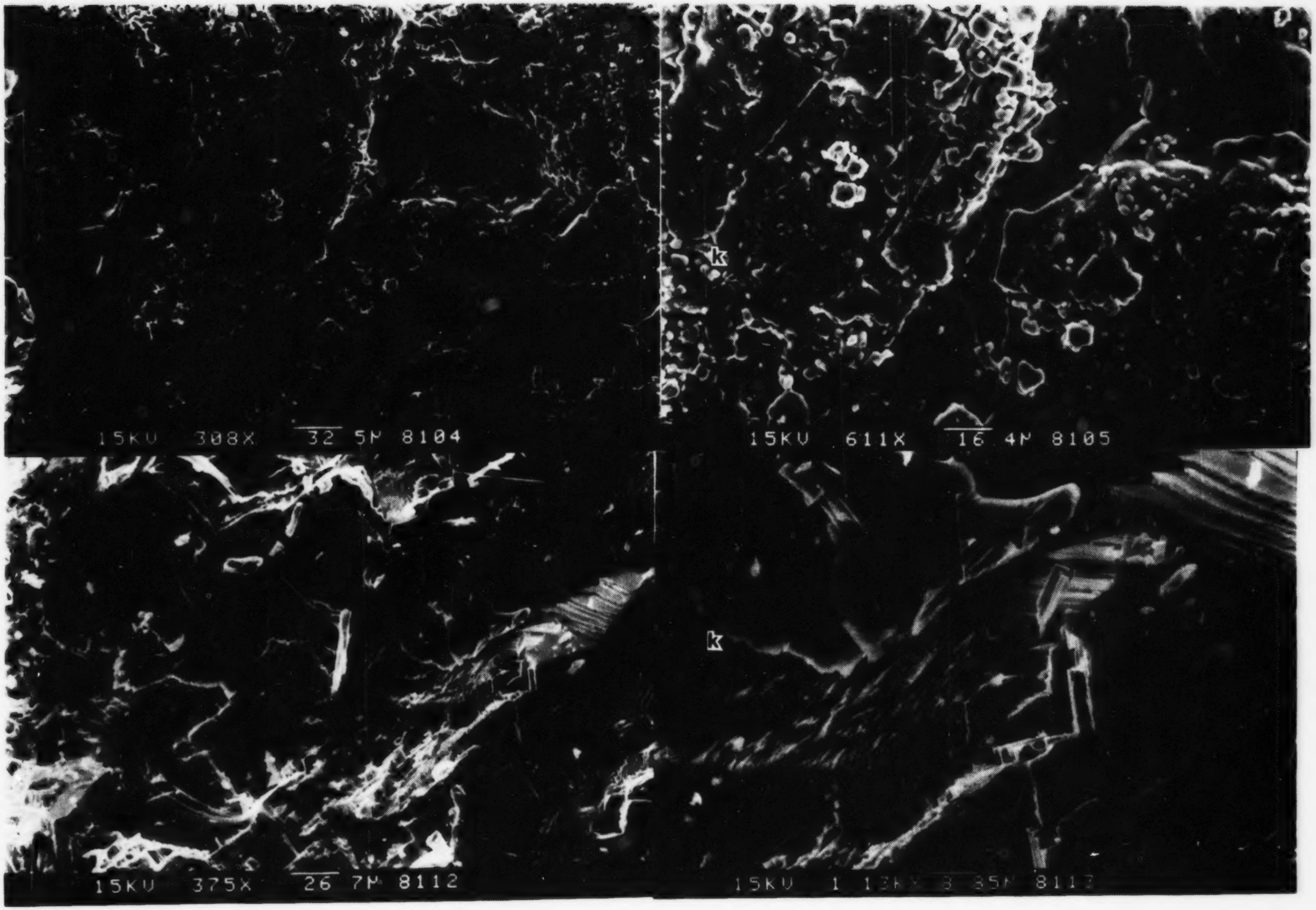
AS 54092
Figure Plate 16
Canola-Arosurf Mg 102 W


15KV 379X 26 4μ 8120
k
15KV 1 14KX 8 77μ 8121

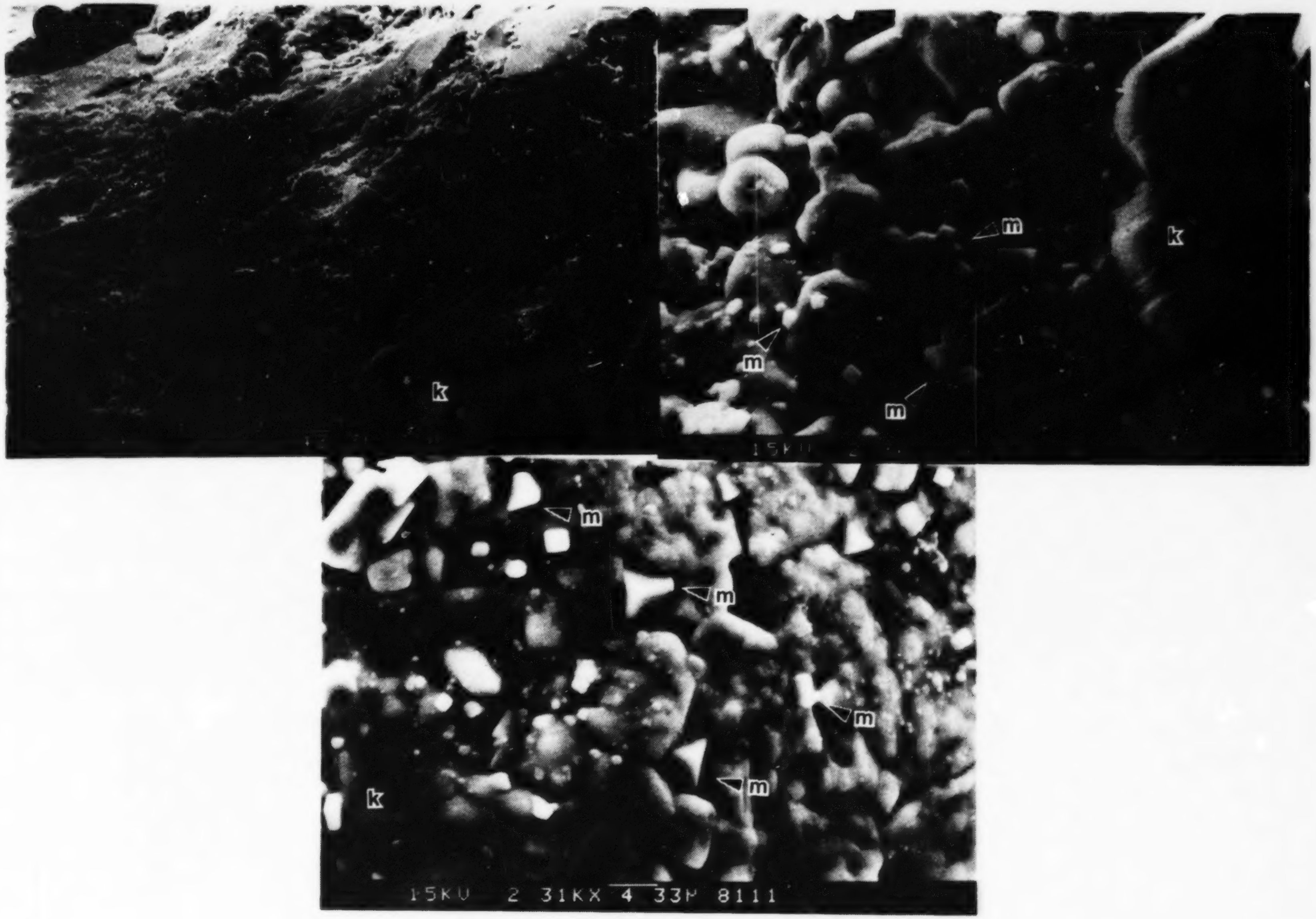
AS 54092
Figure Plate 17
Canola-Arosurf Mg 102 W


m
m
m
m
q
m
k
15KV 0.75KM 13
q
LT= 60 SECS
8108 FE PART 092-21
COUNTS
400
320
240
160
80
0
S I
A U
C L
C
K
F E
F E
A U
0.000
1.000
2.000
3.000
4.000
5.000
6.000
7.000
8.000
9.000
10.000
ENERGY
KeV

AS 54092
Figure Plate 12
Bar Dust


15KU 308X 32 5µ 8104
15KU 611X 16 4µ 8105
k
15KU 375X 26 7µ 8112
k
15KU 1 13KX 8 85µ 8113

AS 54092
Figure Plate 14
Bar Dust


k
m
m
m
k
m
m
m
m
k
15KV 2 31KX 4 33µ 8111

AS 54092
Figure Plate 13
Bar Dust

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